

Supporting Information

Dehydrogenative Cyclization of 2-Arylbenzoic Acid and 2-Arylbenzamide with Hydrogen Evolution in a Photoelectrochemical Cell

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Table of Contents

1. General information.....	2
2. Experimental procedures.....	3-15
2.1. Preparation and characterization of WO₃ photoanodes.....	3-5
2.2. The dehydrogenative cyclization of 2-arylbenzoic acids.....	5-10
2.3. The dehydrogenative cyclization of 2-arylbenzamides.....	10-12
2.4. Free radical capture experiments.....	12-13
2.5. KIE study procedure.....	13-14
2.6. Synthesis of Urolithin A, B, C.....	14-15
3. Copies of NMR spectra.....	17-50

1. General information

The solvents, reagents, and deuterated solvents were purchased from Energy, Aladdin, Adamas, Innochem and Sigma-Aldrich without further purification unless otherwise noted. Chemical shifts were denoted in ppm on the δ scale relative to residual solvent peaks as an internal standard: CHCl_3 (δ 7.28 for ^1H NMR), CDCl_3 (δ 77.0 for ^{13}C NMR), $\text{DMSO-}d_6$ (δ 2.50 for ^1H NMR), $\text{DMSO-}d_6$ (δ 39.5 for ^{13}C NMR). Signal patterns are indicated as s, singlet; d, doublet; t, triplet; m, multiplet; br, broaden peak. Flash column chromatography was performed on silica gel (200-300 mesh). Electrochemical measurements were taken with a CHI760E electrochemical potentiostat (Shanghai Chenhua, China). FTO substrates were purchased from Dalian Heptachroma SolarTech Co., Ltd. (thickness of ~ 2.2 mm, transmittance of $>90\%$, resistance $<15 \Omega \text{ cm}^{-2}$). Before use, the FTO slides were ultrasonically cleaned in deionized water, ethanol, and acetone, respectively. High resolution mass spectra (HRMS) were obtained from Analysis and Testing Center of Dalian University of Technology on Thermo Fisher Scientific Q Exactive Plus instrument. X-ray Diffraction (XRD) was collected with a SmartLab 9KW diffractometer using $\text{Cu K}\alpha$ radiation (154.1 nm). Scanning electron microscopy (SEM) was conducted with the FEI NovaSEM 450 instrument with an accelerating voltage of 5.0 kV. UV-vis diffuse reflectance spectra (DRS) of the samples were measured using a Lambda 750S spectrophotometer. **1a-1y**, **3a-3l** were prepared according to the known procedures reported in the literature¹⁻².

2. Experimental procedures

2.1. Preparation and characterization of WO₃ photoanodes^{3,14}.

Preparation: Firstly, Na₂WO₄ • 2H₂O (1.68 g) was dissolved in 40 ml of distilled water and stirred for 5 min to form a clear solution. 5 ml of concentrated hydrochloric acid was added to the solution, at which point a flocculent precipitate appeared and stirred for 30 min. Finally, 1.8g of anhydrous oxalic acid and 45ml of distilled water were added and stirred for 1h until the solution was clear. The resulting solution was added to a 50 ml PTFE kettle liner, with the conductive glass side of the FTO placed vertically towards the inner wall of the kettle liner, and the kettle liner was placed in an autoclave and reacted for 6 hours at 180°C in an oven. After cooling to room temperature, the excess coating was wiped off and heated in a muffle furnace at 450°C for 2 h. After cooling to room temperature, the electrode was removed and washed with 1 M NaOH and finally with deionized water to obtain the WO₃ electrode.

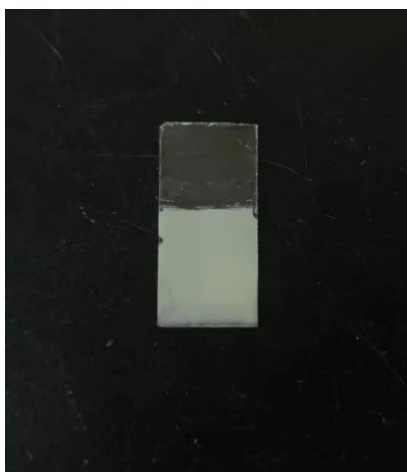


Figure S1. Photograph of WO₃ photoanodes.

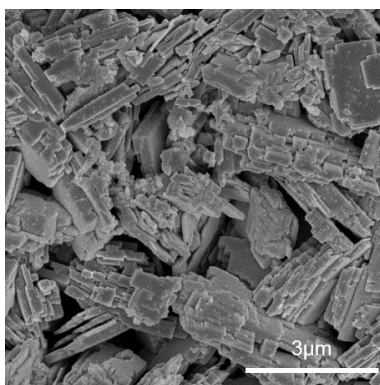


Figure S2. SEM image of WO₃ photoanode after oxidation in 2-phenylbenzoic acid.

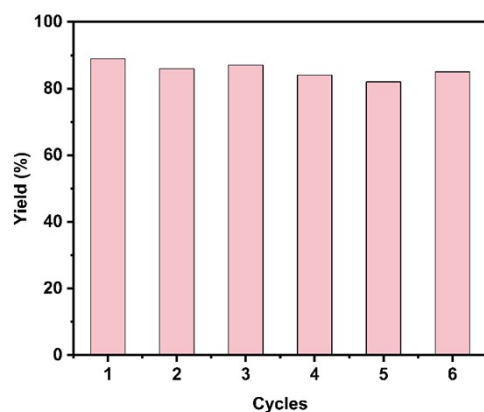


Figure S3. Recycling experiments of WO₃ photoanodes with **1a**.

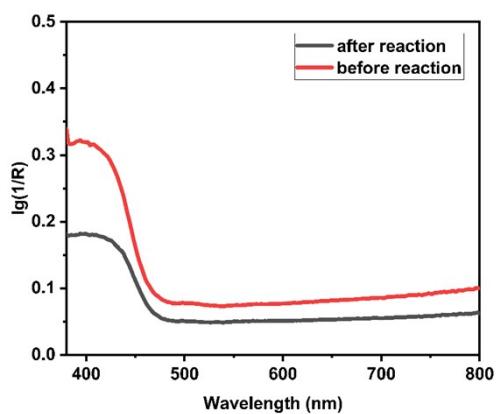


Figure S4. UV-vis adsorption spectra of the WO₃ photoanode before (red) and after (black) reaction.

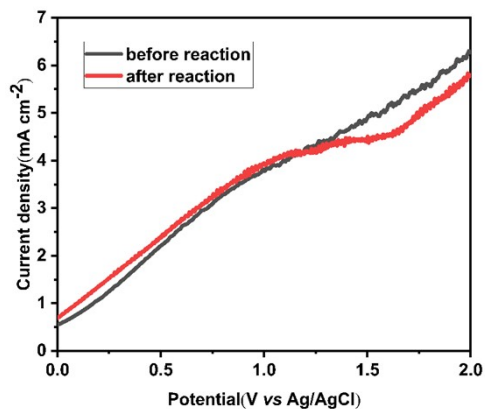


Figure S5. Linear sweep voltammograms of WO₃ photoanode before (gray line) and after (red line) 8 h of PEC electrolysis in 7ml CH₃NO₂ containing 0.2 mmol **1a**, 0.2 mmol TMG and 0.3 mmol TBAClO₄. Scan rate: 10 mV s⁻¹.

IPCE: For a PEC system, the incident monochromatic photon-electron conversion efficiency (IPCE) is used to evaluate the photon-electron conversion efficiency at different wavelengths. According to the equation:

$$IPCE = \frac{|J_{ph}| \times hc/e}{(P_{mono} \times \lambda)}$$

Where J_{ph} is the photocurrent density (mA/cm²), h is the Planck constant (6.62607015×10⁻³⁴ J·s), c is the speed of light (3.0×10⁸ m·s⁻¹), e is the amount of electricity carried by a single electron (1.6×10⁻¹⁹ C), P_{mono} is the incident monochromatic light illumination intensity (100mW/cm²) and λ is the wavelength of incident monochromatic light.

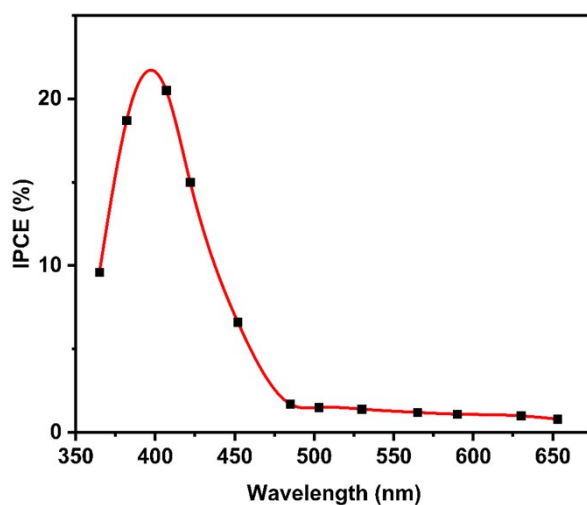
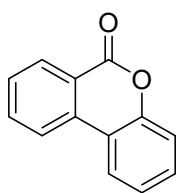


Figure S6. IPCE curves obtained at 1.3 V vs Ag/AgCl

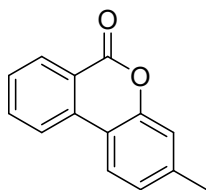
2.2. The dehydrogenative cyclization of 2-arylbenzoic acids

General procedure: The PEC experiments were carried out in an undivided cell equipped with WO₃ as the working electrode (1.0 cm × 1.0 cm), Pt wire as the counter electrode and Ag/AgCl as the reference electrode. Compounds **1** (0.2 mmol) and TMG (0.2 mmol) were dissolved in 7 mL CH₃NO₂ with ⁿBu₄NClO₄ (0.3 mmol) as an electrolyte. The PEC reaction was initiated by blue light (20 W 427 nm) irradiated from the back of the working electrode. Photoelectrochemical electrolysis was performed at a constant potential of +1.3 V vs. Ag/AgCl for 8 h. At the end of the reaction, the mixture was poured onto a silica gel column and the corresponding product was obtained using a mixture of PE: EA=30:1 as eluent.

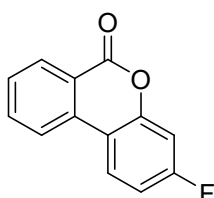
6*H*-Dibenzo[*b,d*]pyran-6-one (**2a**):¹ 35.3mg, 90%yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.38 (d, *J* = 8.0 Hz, 1H), 8.09 (d, *J* = 8.1 Hz, 1H), 8.03 (d, *J* = 7.9 Hz, 1H), 7.82 (t, *J* = 7.7 Hz, 1H), 7.57 (t, *J* = 7.6 Hz, 1H), 7.47 (t, *J* = 7.7 Hz, 1H), 7.41 – 7.29 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 161.2, 151.2, 134.8, 134.7, 130.5, 130.4, 128.9, 124.6, 122.8, 121.7, 121.2, 118.0, 117.7.



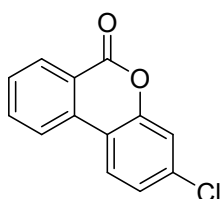
3-Methyl-6*H*-dibenzo[*b,d*]pyran-6-one (**2b**):¹ 33.7mg, 85%yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.38 (dd, *J* = 7.9, 1.4 Hz, 1H), 8.06 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.94 – 7.89 (m, 1H), 7.80 (ddd, *J* = 8.3, 7.3, 1.5 Hz, 1H), 7.55 (ddd, *J* = 8.1, 7.3, 1.1 Hz, 1H), 7.19 – 7.12 (m, 2H), 2.45 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 161.5, 151.3, 141.3, 135.0, 134.8, 130.6, 128.4, 125.7, 122.5, 121.5, 120.9, 117.9, 115.5, 21.5.



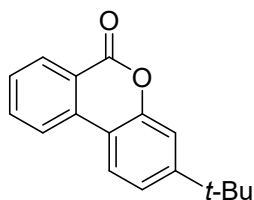
3-Fluoro-6*H*-dibenzo[*b,d*]pyran-6-one (**2c**):¹ 29.1mg, 68%yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.38 (s, 1H), 8.03 (d, *J* = 8.5 Hz, 2H), 7.83 (t, *J* = 7.8 Hz, 1H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.08 (t, *J* = 7.9 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 164.7, 162.2, 160.8, 152.2, 152.1, 135.1, 134.2, 130.7, 128.8, 124.4, 124.3, 121.5, 120.4, 114.6, 114.6, 112.6, 112.3, 105.2, 105.0, 77.4, 77.1, 76.8.



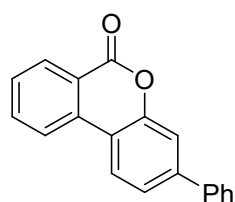
3-Chloro-6*H*-dibenzo[*b,d*]pyran-6-one (**2d**):¹ 36.5mg, 79%yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.39 (dd, *J* = 7.9, 1.4 Hz, 1H), 8.07 (d, *J* = 7.1 Hz, 1H), 7.99 (d, *J* = 8.5 Hz, 1H), 7.85 (ddd, *J* = 8.3, 7.3, 1.5 Hz, 1H), 7.66 – 7.57 (m, 1H), 7.37 (d, *J* = 2.0 Hz, 1H), 7.33 (dd, *J* = 8.5, 2.1 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 160.6, 151.5, 136.0, 135.1, 134.0, 130.7, 129.2, 125.1, 123.8, 121.7, 120.9, 118.0, 116.7.



3-(1,1-Dimethylethyl)-6*H*-dibenzo[*b,d*]pyran-6-one (**2e**):¹ 32.8mg, 65%yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.40 (dd, *J* = 7.9, 1.7 Hz, 1H), 8.10 (d, *J* = 7.6 Hz, 1H), 7.99 (d, *J* = 8.9 Hz, 1H), 7.85 – 7.79 (m, 1H), 7.59 – 7.54 (m, 1H), 7.42 – 7.37 (m, 2H), 1.39 (s, 9H). ¹³C NMR (101 MHz, CDCl₃) δ 161.5, 154.7, 151.2, 134.9, 134.8, 130.5, 128.4, 122.8, 122.0, 121.5, 121.0, 115.4, 114.54, 35.0, 31.1.

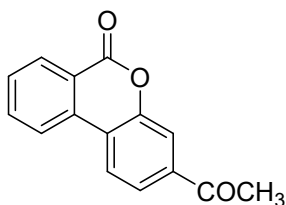


3-Phenyl-6*H*-dibenzo[*b,d*]pyran-6-one (**2f**):⁵ 41.9mg, 77%yield, white solid. ¹H NMR (500 MHz, CDCl₃) δ 8.42 (d, *J* = 7.9 Hz, 1H), 8.16 – 8.09 (m, 2H), 7.85 (t, *J* = 7.7 Hz, 1H), 7.67 (d, *J* = 7.6 Hz, 2H), 7.60 (d, *J* = 6.3 Hz, 3H), 7.51 (t, *J* = 7.7 Hz, 2H), 7.44 (t, *J* = 7.3 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 161.3, 151.7, 143.5, 139.2, 134.9, 134.6,

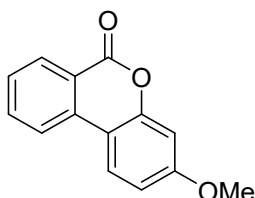


130.7, 129.1, 128.8, 128.3, 127.1, 123.3, 123.2, 121.7, 121.12, 116.9, 115.8.

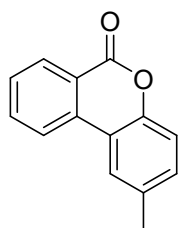
3-Acetyl-6*H*-dibenzo[*b,d*]pyran-6-one (**2g**):⁶ 36.7mg, 77%yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.44 (d, *J* = 7.9 Hz, 1H), 8.18 (t, *J* = 8.8 Hz, 2H), 7.96 – 7.87 (m, 3H), 7.69 (t, *J* = 8.3 Hz, 1H), 2.69 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 196.5, 160.6, 151.1, 138.4, 135.1, 133.6, 130.8, 130.1, 123.9, 123.2, 122.4, 122.0, 121.8, 117.9, 26.3.



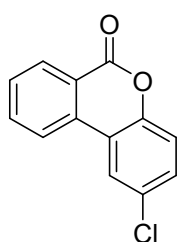
3-Methoxy-6*H*-dibenzo[*b,d*]pyran-6-one (**2h**):¹ 34.8mg, 77%yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.36 (d, *J* = 6.5 Hz, 1H), 8.00 (d, *J* = 8.1 Hz, 1H), 7.94 (d, *J* = 8.8 Hz, 1H), 7.79 (ddd, *J* = 8.3, 7.3, 1.5 Hz, 1H), 7.56 – 7.47 (m, 1H), 6.92 (dd, *J* = 8.8, 2.6 Hz, 1H), 6.87 (d, *J* = 2.6 Hz, 1H), 3.90 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 161.5, 152.6, 135.2, 134.9, 130.5, 127.7, 123.8, 121.1, 120.0, 112.4, 111.1, 101.6, 55.7.



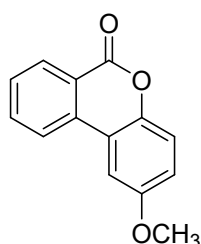
2-Methyl-6*H*-dibenzo[*b,d*]pyran-6-one (**2i**):¹ 33.3mg, 84%yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.33 (d, *J* = 8.0 Hz, 1H), 8.01 (d, *J* = 7.9 Hz, 1H), 7.78 – 7.73 (m, 2H), 7.52 (d, *J* = 7.1 Hz, 1H), 7.24 – 7.17 (m, 2H), 2.42 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 161.3, 149.3, 134.7, 134.7, 134.1, 131.3, 130.5, 128.7, 122.7, 121.6, 121.2, 117.5, 117.4, 21.1.



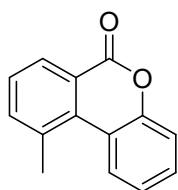
2-Chloro-6*H*-dibenzo[*b,d*]pyran-6-one (**2j**):¹ 34.6mg, 75%yield, white solid. ¹H NMR (500 MHz, CDCl₃) δ 8.39 (d, *J* = 7.8 Hz, 1H), 8.03 (d, *J* = 8.1 Hz, 1H), 7.98 (d, *J* = 2.7 Hz, 1H), 7.85 (t, *J* = 7.7 Hz, 1H), 7.63 (t, *J* = 7.6 Hz, 1H), 7.42 (d, *J* = 8.7 Hz, 1H), 7.30 (d, *J* = 9.0 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 160.6, 149.6, 135.1, 133.5, 130.7, 130.4, 130.1, 129.6, 122.6, 121.8, 121.2, 119.3, 119.2.



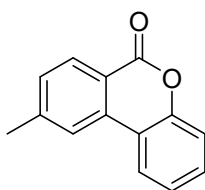
2-Methoxy-6*H*-dibenzo[*b,d*]pyran-6-one (**2k**):⁷ 30.3mg, 67%yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.33 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.98 (d, *J* = 7.3 Hz, 1H), 7.79 – 7.75 (m, 1H), 7.55 (d, *J* = 8.1 Hz, 1H), 7.39 (d, *J* = 3.0 Hz, 1H), 7.22 (d, *J* = 9.0 Hz, 1H), 6.99 (dd, *J* = 8.9, 2.9 Hz, 1H), 3.87 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 161.2, 156.3, 145.5, 134.7, 134.5, 130.5, 128.9, 121.6, 121.2, 118.6, 118.4, 117.1, 106.2, 55.8.



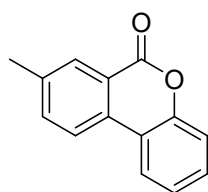
10-Methyl-6*H*-dibenzo[*b,d*]pyran-6-one (**2l**):⁵ 27.4mg, 69%yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.35 (d, *J* = 7.8 Hz, 1H), 8.28 (d, *J* = 8.3 Hz, 1H), 7.63 (d, *J* = 7.5 Hz, 1H), 7.49 – 7.43 (m, 2H), 7.38 (d, *J* = 6.6 Hz, 1H), 7.32 (t, *J* = 7.7 Hz, 1H), 2.88 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 161.7, 151.2, 139.1, 135.1, 133.5, 129.6, 129.1, 128.2, 127.2, 124.0, 122.7, 119.6, 117.9, 25.4.



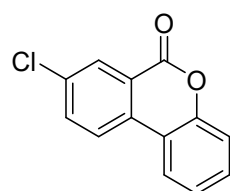
9-Methyl-6*H*-dibenzo[*b,d*]pyran-6-one (**2m**):¹ 34.1mg, 86%yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.19 (d, *J* = 8.0 Hz, 1H), 7.94 (d, *J* = 8.3 Hz, 1H), 7.79 (s, 1H), 7.45 – 7.40 (m, 1H), 7.30 (dd, *J* = 9.9, 7.9 Hz, 3H), 2.51 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 161.2, 151.3, 145.9, 134.6, 130.4, 130.2, 130.1, 124.4, 122.7, 121.8, 118.6, 118.0, 117.6, 22.3.



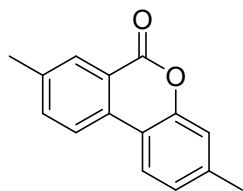
8-Methyl-6*H*-dibenzo[*b,d*]pyran-6-one (**2n**):¹ 37.6mg, 95%yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.13 (s, 1H), 7.95 (dd, *J* = 12.4, 8.1 Hz, 2H), 7.58 (dd, *J* = 8.3, 2.3 Hz, 1H), 7.42 (ddd, *J* = 8.4, 7.0, 1.5 Hz, 1H), 7.33 – 7.27 (m, 2H), 2.46 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 161.3, 150.9, 139.2, 136.0, 132.1, 130.2, 129.9, 124.5, 122.5, 121.6, 121.0, 118.1, 117.6, 21.3.



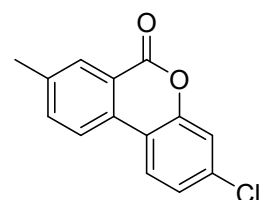
8-Chloro-6*H*-dibenzo[*b,d*]pyran-6-one (**2o**):⁷ 41mg, 89%yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.32 (d, *J* = 2.4 Hz, 1H), 8.04 (d, *J* = 8.6 Hz, 1H), 7.99 (d, *J* = 6.8 Hz, 1H), 7.76 (dd, *J* = 8.6, 2.3 Hz, 1H), 7.50 (t, *J* = 7.8 Hz, 1H), 7.35 (t, *J* = 7.4 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 160.0, 151.0, 135.0, 133.2, 130.9, 130.0, 124.9, 123.4, 122.8, 122.5, 117.9, 117.2.



3,8-Dimethyl-6*H*-dibenzo[*b,d*]pyran-6-one (**2p**):¹ 33.6mg, 75%yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.18 (s, 1H), 7.97 (d, *J* = 8.3 Hz, 1H), 7.90 (d, *J* = 8.0 Hz, 1H), 7.62 (d, *J* = 8.3 Hz, 1H), 7.15 (d, *J* = 9.9 Hz, 2H), 2.50 (s, 3H), 2.46 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 161.7, 151.0, 140.7, 138.6, 136.0, 132.5, 130.3, 125.6, 122.3, 121.4, 120.7, 117.8, 115.6, 21.4, 21.3.

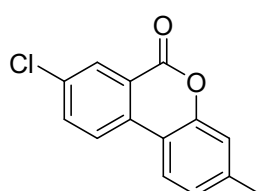


3-Chloro-8-methyl-6*H*-dibenzo[*b,d*]pyran-6-one (**2q**):¹² 36.7mg, 75%yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.18 (s, 1H), 7.97 – 7.91 (m, 2H), 7.65 (dd, *J* = 8.3, 2.3 Hz, 1H), 7.35 (d, *J* = 2.1 Hz, 1H),

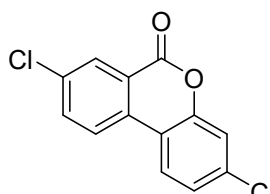


7.30 (dd, $J = 8.5, 2.1$ Hz, 1H), 2.51 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 160.8, 151.2, 139.7, 136.3, 135.3, 131.4, 130.5, 124.9, 123.6, 121.7, 120.7, 117.8, 116.9, 21.4.

8-Chloro-3-methyl-6*H*-dibenzo[*b,d*]pyran-6-one (**2r**):⁷ 39.6mg, 81%yield, white solid.

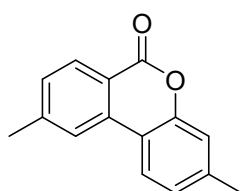


^1H NMR (400 MHz, CDCl_3) δ 8.35 (d, $J = 2.3$ Hz, 1H), 8.03 (d, $J = 8.6$ Hz, 1H), 7.89 (d, $J = 8.5$ Hz, 1H), 7.76 (dd, $J = 8.6, 2.4$ Hz, 1H), 7.20 – 7.15 (m, 2H), 2.47 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 160.3, 151.1, 141.8, 135.1, 134.4, 133.5, 130.0, 126.0, 123.2, 122.5, 122.1, 118.0, 114.7, 21.5.



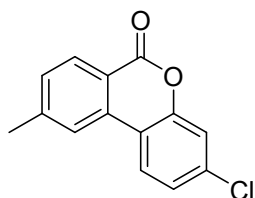
3,8-Dichloro-6*H*-dibenzo[*b,d*]pyran-6-one (**2s**): unknown compound, 37.6mg, 71%yield, white solid, m.p. 224.6-225.9 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.36 (d, $J = 2.3$ Hz, 1H), 8.02 (d, $J = 8.6$ Hz, 1H), 7.95 (d, $J = 8.5$ Hz, 1H), 7.80 (dd, $J = 8.6, 2.3$ Hz, 1H), 7.41 – 7.33 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.4, 151.4, 136.4, 135.4, 135.4, 132.5, 130.2, 125.3, 123.8, 123.4, 122.2, 118.1, 116.0.

3,9-Dimethyl-6*H*-dibenzo[*b,d*]pyran-6-one (**2t**):¹ 34.0mg, 76%yield, white solid. ^1H



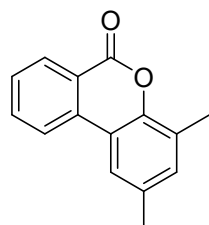
NMR (400 MHz, CDCl_3) δ 8.20 (d, $J = 8.0$ Hz, 1H), 7.84 (d, $J = 8.5$ Hz, 1H), 7.78 (s, 1H), 7.31 (d, $J = 8.1$ Hz, 1H), 7.10 (d, $J = 6.0$ Hz, 2H), 2.52 (s, 3H), 2.43 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 161.5, 151.3, 146.2, 141.0, 134.8, 130.4, 129.6, 125.5, 122.4, 121.5, 118.3, 117.7, 115.4, 22.3, 21.4.

3-Chloro-9-methyl-6*H*-dibenzo[*b,d*]pyran-6-one (**2u**):¹ 32.8mg, 67%yield, white solid.



^1H NMR (500 MHz, CDCl_3) δ 8.21 (d, $J = 8.1$ Hz, 1H), 7.91 (d, $J = 8.5$ Hz, 1H), 7.78 (s, 1H), 7.38 (d, $J = 8.1$ Hz, 1H), 7.31 – 7.25 (m, 2H), 2.55 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 160.6, 151.6, 146.3, 135.7, 133.8, 130.6, 130.4, 124.9, 123.7, 121.8, 118.3, 117.8, 116.7, 22.3.

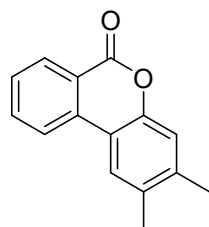
2,4-Dimethyl-6*H*-dibenzo[*b,d*]pyran-6-one (**2v**):¹ 26.0mg, 58%yield, white solid. ^1H



NMR (400 MHz, CDCl_3) δ 8.38 (d, $J = 9.5$ Hz, 1H), 8.05 (d, $J = 8.9$ Hz, 1H), 7.78 (t, $J = 6.9$ Hz, 1H), 7.63 (s, 1H), 7.54 (t, $J = 7.6$ Hz, 1H), 7.12 (s, 1H), 2.44 (s, 3H), 2.41 (s, 3H). ^{13}C NMR (101 MHz,

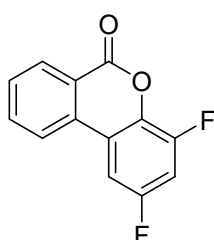
CDCl₃) δ 161.4, 147.7, 135.1, 134.6, 133.4, 132.8, 130.4, 128.4, 126.6, 121.8, 121.0, 120.3, 117.2, 21.1, 15.9.

2,3-Dimethyl-6*H*-dibenzo[*b,d*]pyran-6-one (**2w**):¹⁰ 31.8mg, 71%yield, white solid. ¹H



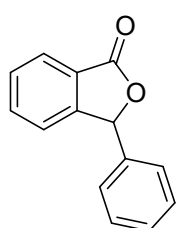
NMR (400 MHz, CDCl₃) δ 8.40 (d, *J* = 6.5 Hz, 1H), 8.09 (d, *J* = 8.1 Hz, 1H), 7.80 (d, *J* = 7.0 Hz, 2H), 7.56 (d, *J* = 7.1 Hz, 1H), 7.15 (s, 1H), 2.37 (d, *J* = 5.6 Hz, 6H). ¹³C NMR (126 MHz, CDCl₃) δ 161.6, 149.5, 140.0, 135.0, 134.7, 133.1, 130.5, 128.2, 123.1, 121.4, 119.5, 118.2, 115.4, 20.0, 19.5.

2,4-Difluoro-6*H*-dibenzo[*b,d*]pyran-6-one (**2x**):⁶ 14.9mg, 32%yield, white solid. ¹H



NMR (400 MHz, CDCl₃) δ 8.45 (dd, *J* = 7.9, 1.9 Hz, 1H), 8.04 (d, *J* = 8.1 Hz, 1H), 7.93 – 7.87 (m, 1H), 7.72 – 7.67 (m, 1H), 7.57 – 7.51 (m, 1H), 7.07 (ddd, *J* = 9.8, 8.1, 2.8 Hz, 1H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 159.3, 157.0, 156.8, 151.1, 151.0, 148.6, 148.5, 136.3, 136.0, 133.4, 130.9, 130.3, 123.9, 121.2, 121.0, 120.9, 106.5, 106.2, 106.2, 106.0, 105.7, 105.7.

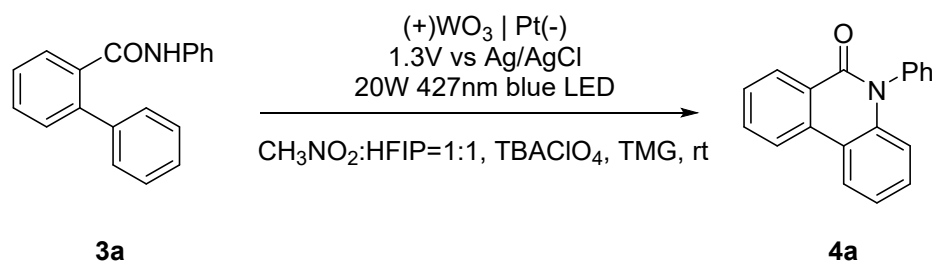
3-phenylisobenzofuran-1(3*H*)-one (**2z**):¹ 7.6mg, 18%yield, white solid. ¹H NMR (400



MHz, CDCl₃) δ 7.99 (d, *J* = 7.6 Hz, 1H), 7.68 (t, *J* = 7.5 Hz, 1H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.43 – 7.39 (m, 3H), 7.36 (d, *J* = 7.6 Hz, 1H), 7.30 (dd, *J* = 6.7, 3.1 Hz, 2H), 6.43 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 171.96, 170.51, 149.71, 136.47, 134.36, 129.40, 129.34, 129.01, 127.01, 125.70, 125.61, 122.89, 82.76.

2.3. The dehydrogenative cyclization of 2-arylbenzamides

Table S1. Details of reaction condition optimization



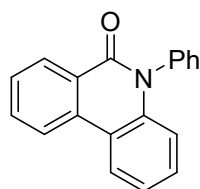
entry	deviation from the standard conditions	yield(%) ^b
1	None	93%
2	pyridine instead of TMG, CH ₃ NO ₂ instead of mixed solvent	56%
3	DMAP instead of TMG, CH ₃ NO ₂ instead of mixed solvent	45%
4	2,6-lutidine instead of TMG, CH ₃ NO ₂ instead of mixed solvent	44%
5	DBU instead of TMG, CH ₃ NO ₂ instead of mixed solvent	62%

6	Na ₂ CO ₃ instead of TMG, CH ₃ NO ₂ instead of mixed solvent	52%
7	CH ₃ COONa instead of TMG, CH ₃ NO ₂ instead of mixed solvent	48%
8	CH ₃ CN instead of CH ₃ NO ₂ : HFIP = 1 : 1	trace
9	HFIP instead of CH ₃ NO ₂ : HFIP = 1 : 1	88%
10	CH ₃ NO ₂ instead of CH ₃ NO ₂ : HFIP = 1 : 1	65%
11	no TMG	42%
12	no irradiation	nd
13	no electrolyte	nd

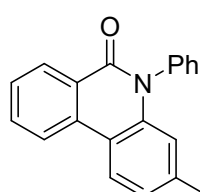
^aStandard conditions: undivided cell, WO₃ photoanode (1.0 cm × 1.0 cm), Pt cathode, **3a** (0.2 mmol), TBAClO₄ (0.3 mmol), CH₃NO₂ (3.5 ml), HFIP (3.5 ml), TMG (0.2 mmol) at room temperature for 8 h under 20W 427nm LED. ^bIsolated yield.

General procedure: The PEC experiments were carried out in an undivided cell equipped with WO₃ as the working electrode (1.0 cm × 1.0 cm), Pt wire as the counter electrode and Ag/AgCl as the reference electrode. 2-Arylbenzamides **3** (0.2 mmol) and TMG (0.2 mmol) were dissolved in 3.5 mL CH₃NO₂ and 3.5ml HFIP with ⁿBu₄NClO₄ (0.3 mmol) as an electrolyte. The PEC reaction was initiated by blue light (20 W 427 nm) irradiated from the back of the working electrode. Photoelectrochemical electrolysis was performed at a constant potential of +1.3 V vs. Ag/AgCl for 8 h. At the end of the reaction, the mixture was poured onto a silica gel column and the corresponding product was obtained using a mixture of PE:EA=50:1 as eluent.

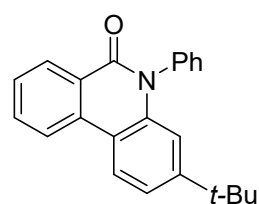
5-Phenyl-6(5*H*)-phenanthridinone (**4a**):¹¹ 50.5mg, %yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.60 (d, *J* = 8.0 Hz, 1H), 8.40 – 8.30 (m, 2H), 7.88 – 7.81 (m, 1H), 7.69 – 7.55 (m, 4H), 7.41 – 7.29 (m, 4H), 6.73 (dd, *J* = 7.9, 1.8 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 161.8, 139.2, 138.3, 134.0, 132.9, 130.3, 129.1, 129.1, 129.1, 128.8, 128.2, 125.9, 123.0, 122.7, 121.8, 119.1, 117.1.



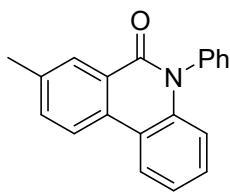
3-Methyl-5-phenyl-6(5*H*)-phenanthridinone (**4b**):¹³ 47.4mg, 83%yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.58 (d, *J* = 9.5 Hz, 1H), 8.31 (d, *J* = 8.1 Hz, 1H), 8.20 (d, *J* = 8.3 Hz, 1H), 7.84 – 7.79 (m, 1H), 7.68 – 7.56 (m, 4H), 7.37 (d, *J* = 7.0 Hz, 2H), 7.13 (d, *J* = 8.1 Hz, 1H), 6.52 (s, 1H), 2.32 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 162.0, 139.6, 139.2, 138.4, 134.1, 132.8, 130.3, 129.2, 129.0, 128.8, 127.7, 125.5, 124.0, 123.0, 121.6, 117.2, 116.7, 22.4.



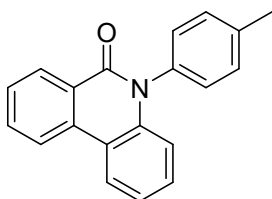
3-(1,1-Dimethylethyl)-5-phenyl-6(5*H*)-phenanthridinone (**4c**):¹¹ 57.0mg, 87%yield, white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.58 (dd, *J* = 8.0, 1.5 Hz, 1H), 8.34 (d, *J* = 8.8 Hz, 1H), 8.25 (d, *J* = 8.5 Hz, 1H), 7.85 – 7.79 (m, 1H), 7.69 – 7.55 (m, 4H), 7.43 – 7.33 (m, 3H), 6.72 (d, *J* = 1.9 Hz, 1H), 1.22 (s, 9H). ¹³C NMR (101 MHz, CDCl₃) δ 162.0, 152.7, 139.1, 138.4, 134.1, 132.8, 130.2, 129.1, 129.0, 128.8, 127.7, 125.6, 122.7, 121.7, 120.3, 116.7, 113.9, 34.9, 31.0.



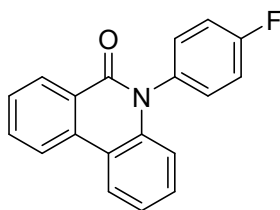
8-Methyl-5-phenyl-6(5*H*)-phenanthridinone (**4d**): unknown compound, 48.5mg, 85%yield, white solid, m.p.191.7-193.1°C. ¹H NMR (500 MHz, CDCl₃) δ 8.38 (s, 1H), 8.30 (dd, *J* = 5.7, 3.9 Hz, 1H), 8.26 (d, *J* = 8.2 Hz, 1H), 7.67 – 7.62 (m, 3H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.36 (d, *J* = 7.2 Hz, 2H), 7.31 – 7.29 (m, 2H), 6.70 (dd, *J* = 6.6, 3.0 Hz, 1H), 2.56 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 161.8, 138.9, 138.5, 138.4, 134.2, 131.6, 130.2, 129.1, 128.8, 128.6, 125.8, 122.8, 122.6, 121.8, 119.2, 117.0, 21.4.



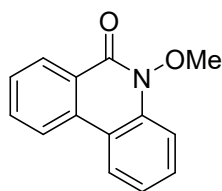
5-(4-Methylphenyl)-6(5*H*)-phenanthridinone (**4e**):¹¹ 54.2mg, 95%yield, white solid. ¹H NMR (500 MHz, CDCl₃) δ 8.59 (d, *J* = 9.6 Hz, 1H), 8.35 (dd, *J* = 17.7, 8.9 Hz, 2H), 7.84 (t, *J* = 7.7 Hz, 1H), 7.64 (t, *J* = 7.6 Hz, 1H), 7.44 (d, *J* = 8.2 Hz, 2H), 7.32 (td, *J* = 7.1, 1.8 Hz, 2H), 7.24 (d, *J* = 8.2 Hz, 2H), 6.77 (d, *J* = 9.8 Hz, 1H), 2.51 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 161.8, 139.4, 138.7, 135.6, 134.0, 132.8, 130.9, 129.1, 128.8, 128.1, 125.9, 123.0, 122.6, 121.8, 119.1, 117.1, 21.4.



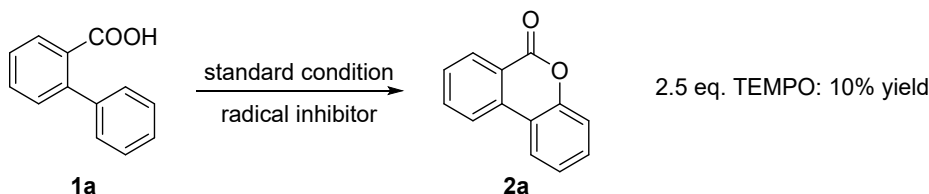
5-(4-Fluorophenyl)-6(5*H*)-phenanthridinone (**4f**):¹¹ 53.8mg, 93%yield, white solid. ¹H NMR (500 MHz, CDCl₃) δ 8.58 (d, *J* = 8.1 Hz, 1H), 8.36 (dd, *J* = 13.3, 7.8 Hz, 2H), 7.85 (t, *J* = 6.9 Hz, 1H), 7.65 (t, *J* = 7.6 Hz, 1H), 7.37 – 7.31 (m, 6H), 6.72 (d, *J* = 7.8 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 163.6, 161.9, 161.6, 139.2, 134.0, 133.0, 131.0, 130.9, 129.2, 129.1, 128.3, 125.8, 123.1, 122.8, 121.9, 119.1, 117.4, 117.2, 116.9.



5-Methoxy-6(5*H*)-phenanthridinone (**4g**):⁹ 26.6mg, 59%yield, yellow solid. ¹H NMR (500 MHz, CDCl₃) δ 8.59 (d, *J* = 7.9 Hz, 1H), 8.30 (dd, *J* = 8.1, 3.7 Hz, 2H), 7.81 (t, *J* = 7.6 Hz, 1H), 7.71 (d, *J* = 9.6 Hz, 1H), 7.62 (q, *J* = 7.5, 7.0 Hz, 2H), 7.38 (t, *J* = 7.6 Hz, 1H), 4.17 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 157.3, 135.9, 133.0, 132.7, 130.0, 128.6, 128.1, 126.4, 123.2, 122.0, 118.6, 112.7, 62.7.



2.4. Free radical capture experiments



Based on the general procedure, TEMPO (2.5 equiv) were added to the reaction. When TEMPO (2,2,6,6-tetramethylpiperidine-1-oxyl) was added to the reaction system, the yield of product **2a** was reduced to 10%.

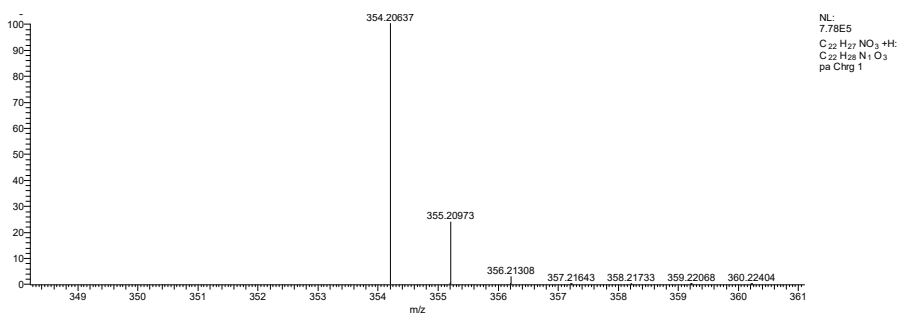
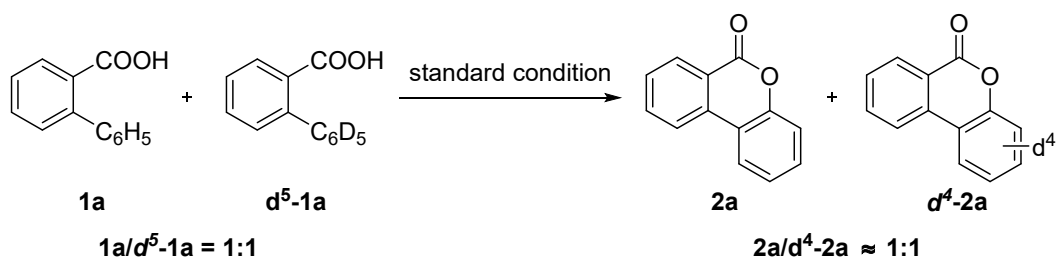


Figure S7.The HRMS of adduct of TEMPO and **2a**

2.5. KIE study procedure



The PEC experiments were carried out in an undivided cell equipped with WO_3 as the working electrode ($1.0 \text{ cm} \times 1.0 \text{ cm}$), Pt wire as the counter electrode and Ag/AgCl as the reference electrode. The mixed substrate **d⁵-1a** and **1a** (1/1, 0.2 mmol) and TMG (0.2 mmol) were dissolved in 7 mL CH_3NO_2 with $n\text{Bu}_4\text{NClO}_4$ (0.3 mmol) as an electrolyte. The PEC reaction was initiated by blue light (20 W 427 nm) irradiated from the back of the working electrode. Photoelectrochemical electrolysis was performed at a constant potential of +1.3 V vs. Ag/AgCl for 20 min. Then the solvent was removed with a rotary evaporator and the residue was purified by column chromatography on silica gel to afford the desired product. The ratio of two kinds of products was determined by ^1H NMR spectrum.

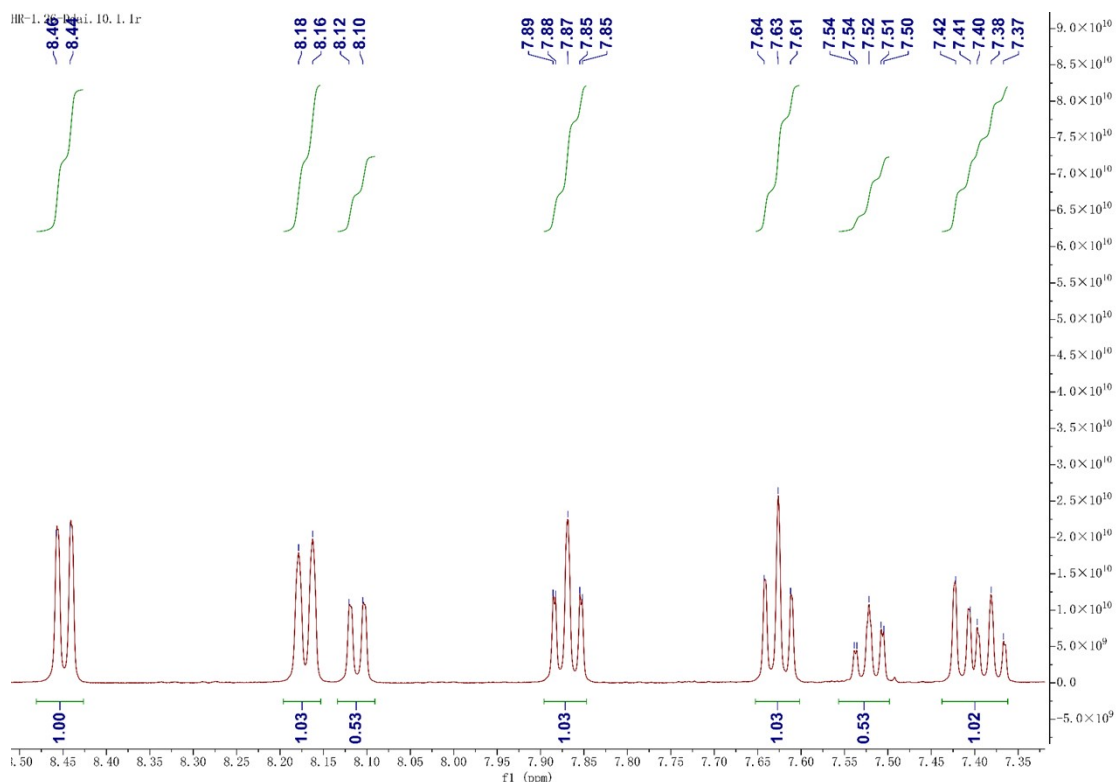
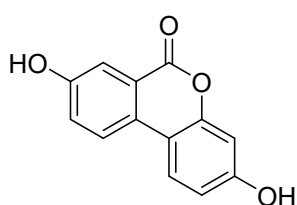


Figure S8.The ^1H NMR of the product **2a/d⁴-2a**

2.6. Synthesis of Urolithin A, B, C

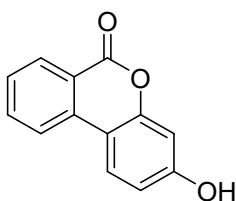
The cyclised products were prepared using standard procedures followed by demethylation with reference to the literature.³

Urolithin A, 17.8mg, 65%yield, yellow solid. ^1H NMR (500 MHz, DMSO-*d*₆) δ 10.20



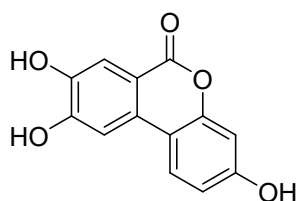
(s, 2H), 8.02 (dd, $J = 41.5, 8.9$ Hz, 2H), 7.51 (s, 1H), 7.31 (d, $J = 8.9$ Hz, 1H), 6.80 (d, $J = 8.7$ Hz, 1H), 6.72 (s, 1H). ^{13}C NMR (126 MHz, DMSO-*d*₆) δ 161.1, 159.1, 157.5, 151.4, 127.4, 124.6, 124.2, 123.9, 120.6, 114.0, 113.5, 110.3, 103.3.

Urolithin B, 22.9mg, 70%yield, yellow solid. ^1H NMR (500 MHz, DMSO-*d*₆) δ 10.36



(s, 1H), 8.32 – 8.09 (m, 3H), 7.86 (d, $J = 7.5$ Hz, 1H), 7.54 (d, $J = 5.3$ Hz, 1H), 6.91 – 6.69 (m, 2H). ^{13}C NMR (126 MHz, DMSO-*d*₆) δ 161.1, 160.3, 152.6, 135.7, 135.6, 130.1, 128.1, 125.3, 122.1, 119.4, 113.6, 109.8, 103.4.

Urolithin C, 13.7mg, 74%yield, red solid. ^1H NMR (500 MHz, DMSO-*d*₆) δ 10.43 (s,



1H), 10.16 (s, 1H), 10.06 (s, 1H), 7.85 (d, $J = 8.8$ Hz, 1H), 7.50 (s, 1H), 7.44 (s, 1H), 6.79 (dd, $J = 8.7, 2.4$ Hz, 1H), 6.70 (d, $J = 2.3$ Hz, 1H). ^{13}C NMR (126 MHz, DMSO-*d*₆) δ 160.8,

159.0, 153.9, 151.9, 146.6, 129.7, 124.2, 114.7, 113.3, 111.3, 110.3, 107.3, 103.3.

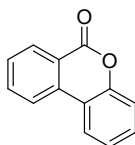
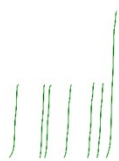
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13. Verma, A.; Singh Banjara, L.; Meena, R.; Kumar, S. Transition-Metal-Free Synthesis of N-Substituted Phenanthridinones and Spiro-isoindolinones: C(sp²)-N and C(sp²)-O Coupling through Radical Pathway. *Asian J. Org. Chem.* **2020**, *9*, 105-110.
14. Chen, Y.; He, Y.; Gao, Y.; Xue, J.; Qu, W.; Xuan, J.; Mo, Y. *Science*, **2024**, *384*, 670-676.

3. Copies of NMR spectra

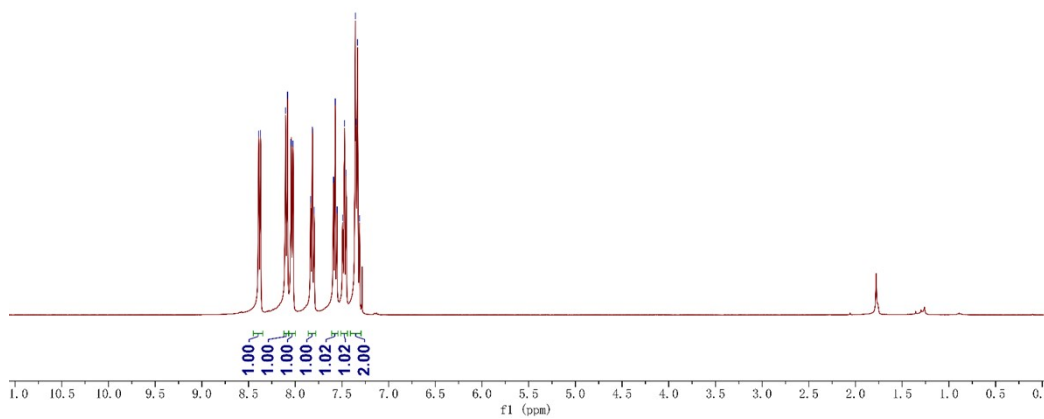
pdata/1

8.39
8.37
8.10
8.08
8.04
8.03
7.84
7.82
7.80
7.59
7.57
7.55
7.49
7.47
7.46
7.36
7.35
7.33
7.31



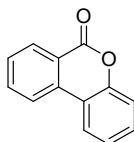
2a

¹H NMR (400 MHz, CDCl₃)



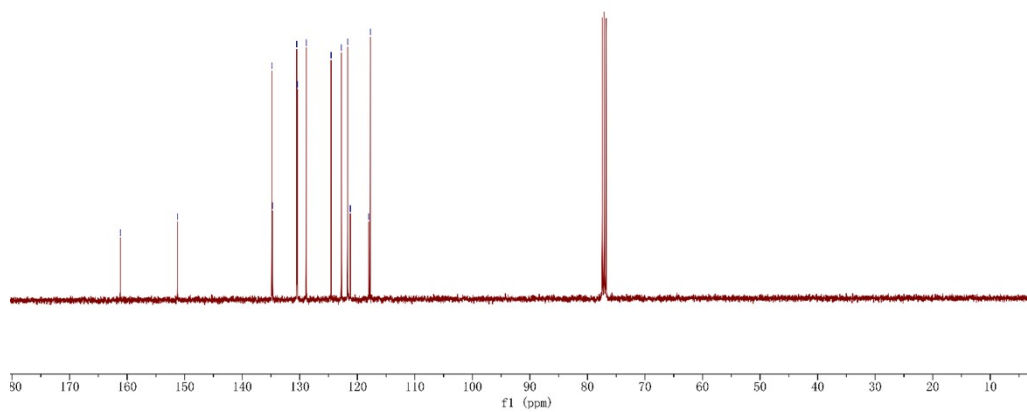
HR-2a-C. 10. 1. 1r

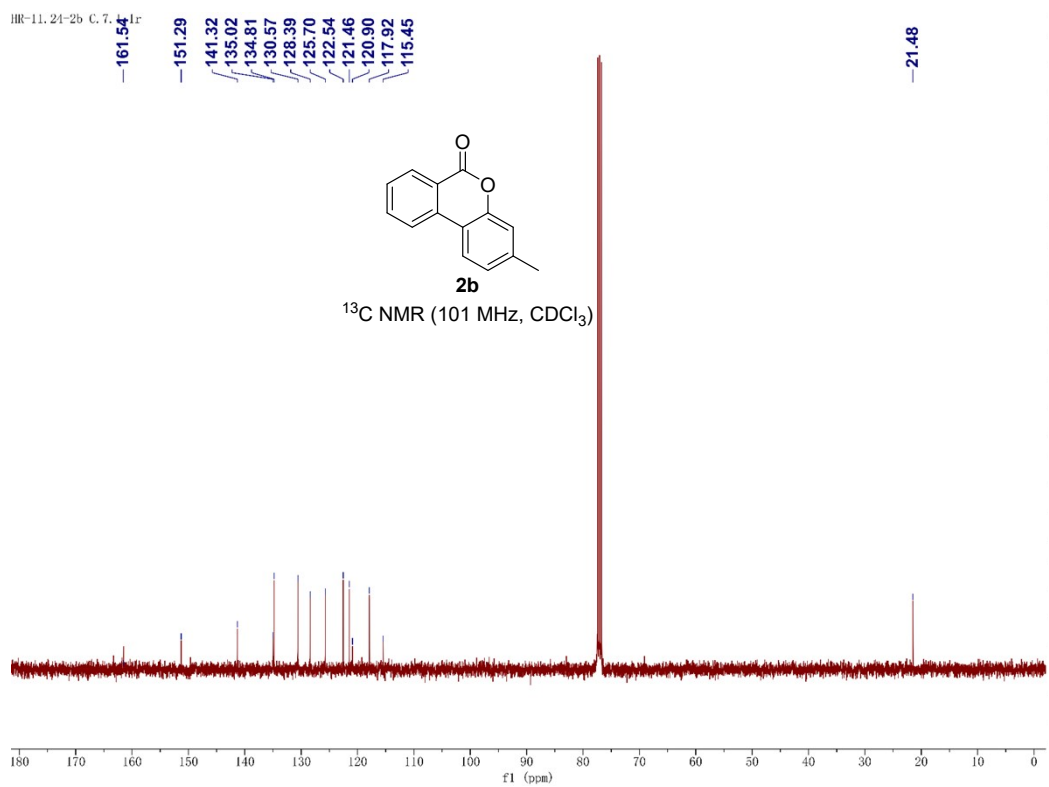
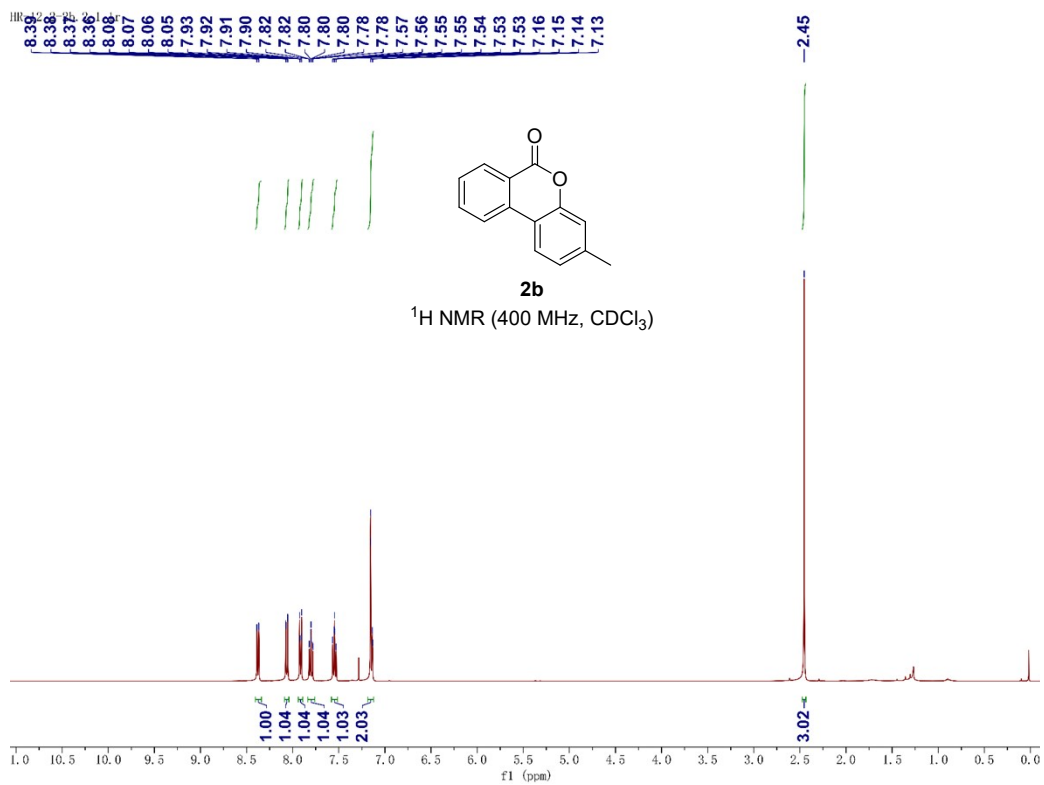
161.17
151.24
134.84
134.72
130.52
130.43
128.87
124.56
122.76
121.68
121.20
117.99
117.73



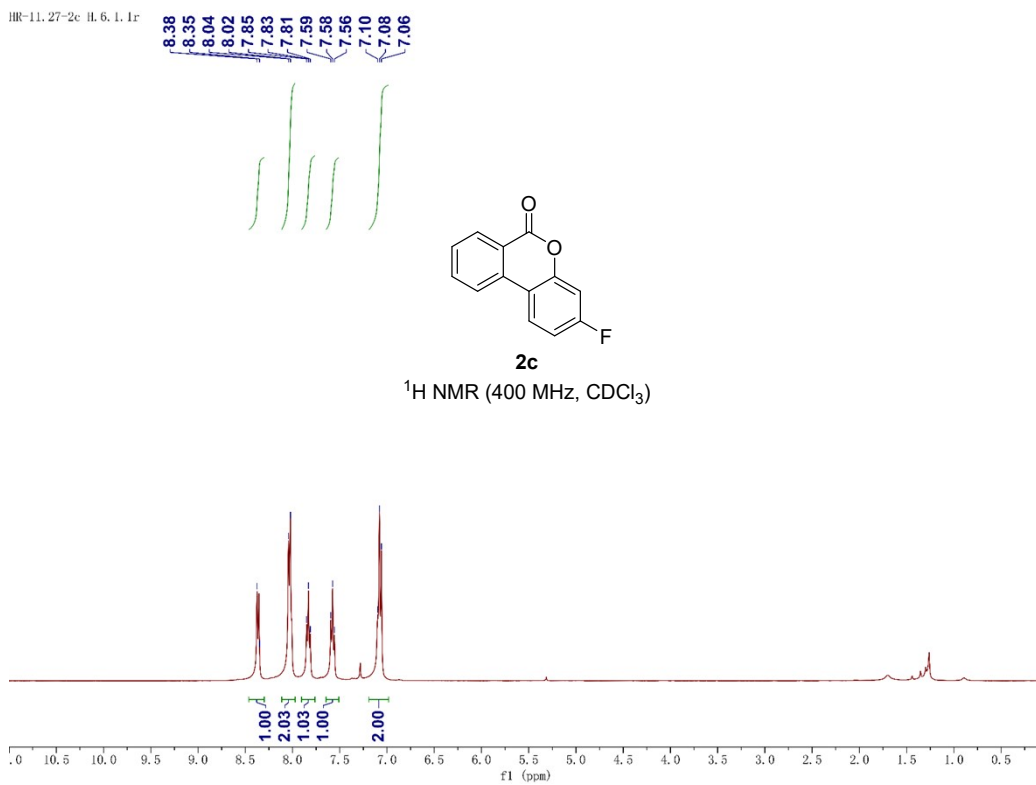
2a

¹³C NMR (101 MHz, CDCl₃)

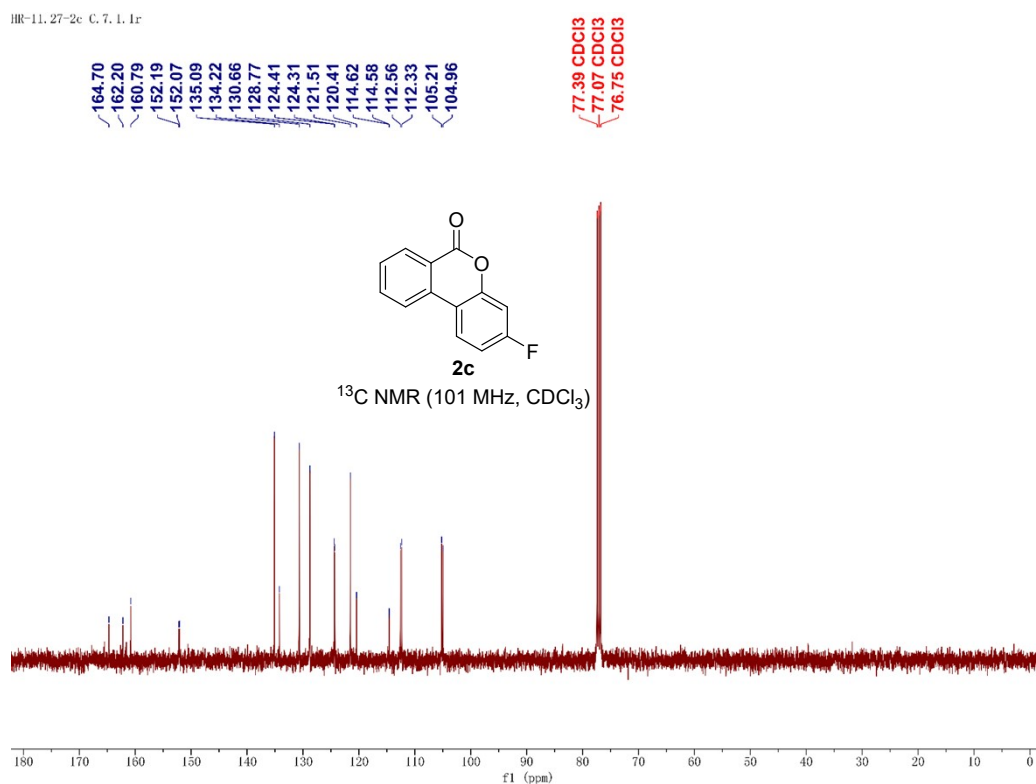




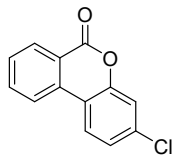
HR-11.27-2c H. 6. 1. 1r



HR-11.27-2c C. 7. 1. 1r

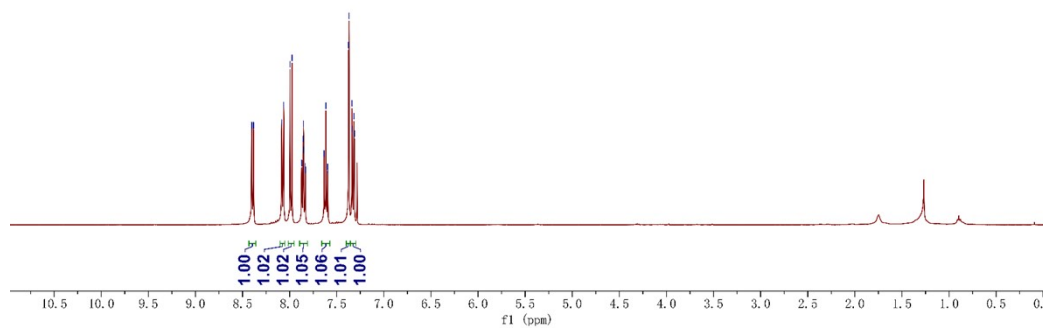


HR-11-
8.40
8.38
8.36
8.08
8.06
8.00
7.97
7.87
7.86
7.85
7.84
7.83
7.64
7.63
7.61
7.60
7.59
7.37
7.34
7.33
7.32
7.31



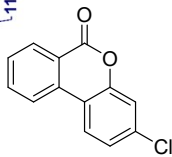
2d

¹H NMR (400 MHz, CDCl₃)



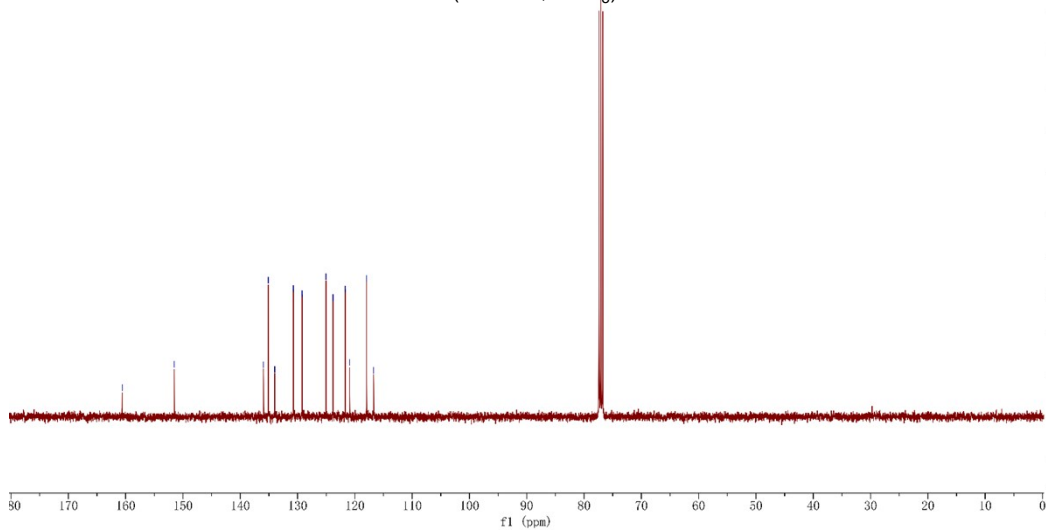
HR-11-28-2d C.7.
160.59
151.50

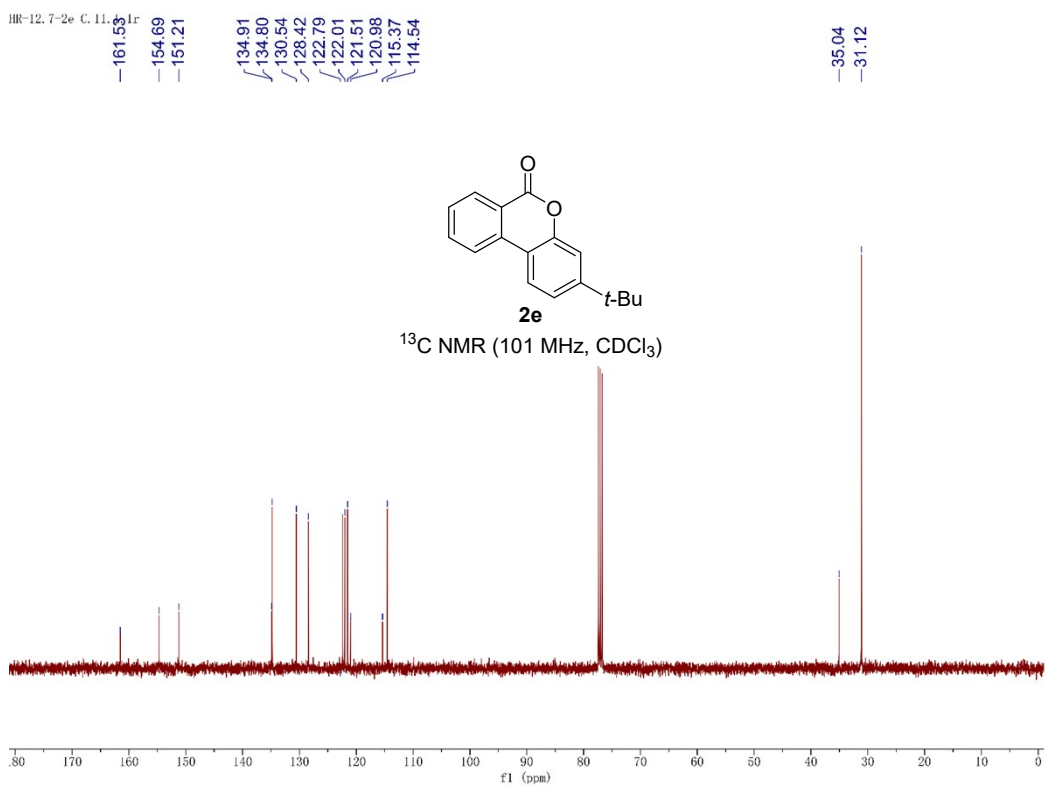
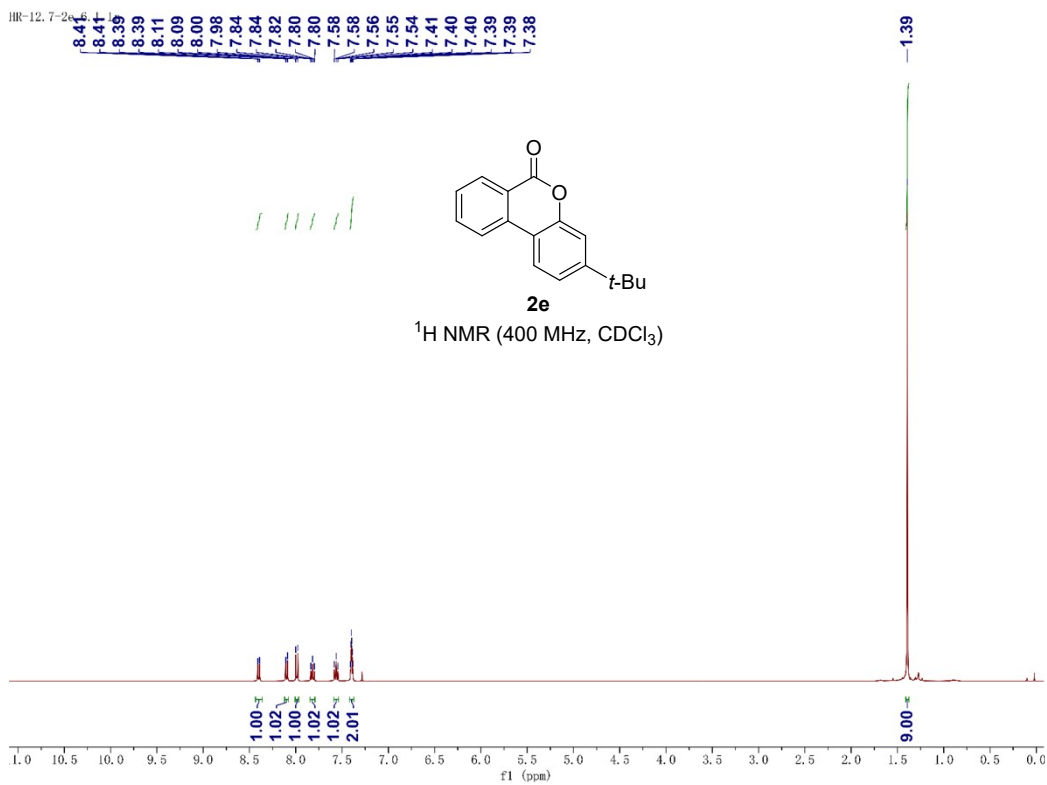
135.94
133.99
130.74
129.22
125.05
123.82
121.69
120.90
117.96
116.72



2d

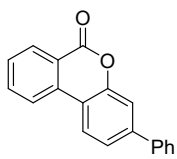
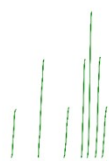
¹³C NMR (101 MHz, CDCl₃)





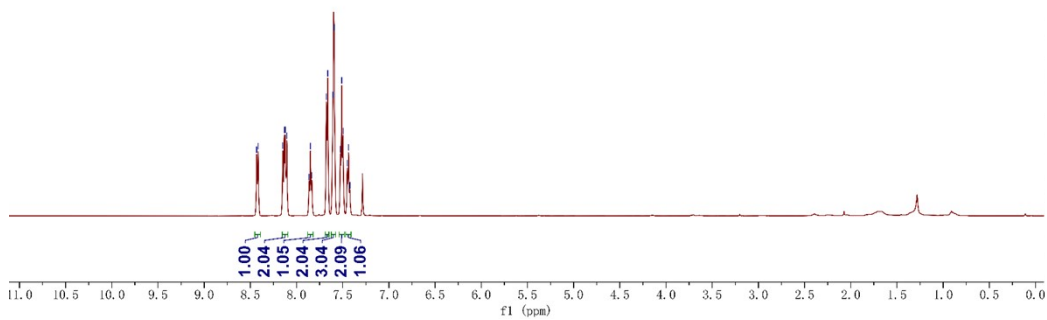
hr-3.23-2f h. 10.

8.43
8.42
8.15
8.13
8.12
8.11
7.87
7.85
7.83
7.68
7.66
7.61
7.59
7.53
7.51
7.50
7.45
7.44
7.42



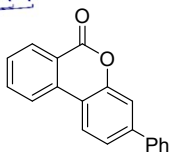
2f

¹H NMR (500 MHz, CDCl₃)



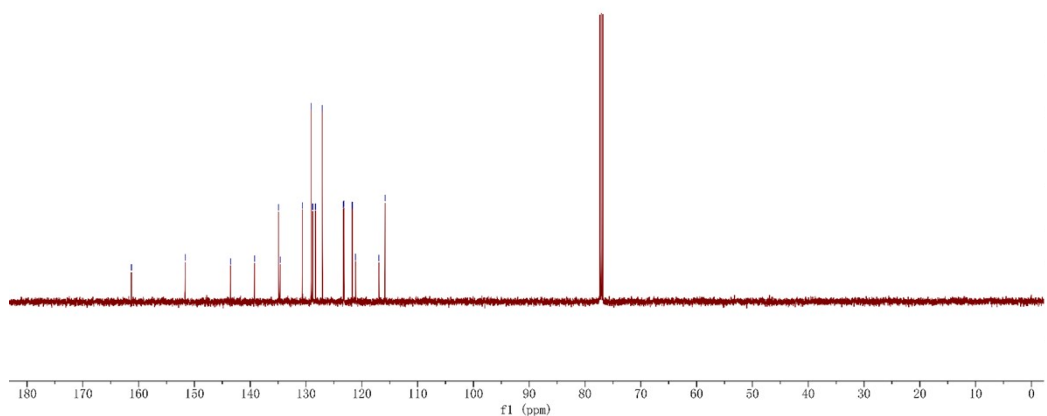
hr-3.23-2f i. 12. f16

161.26
151.65
143.51
139.23
134.91
134.63
130.65
129.08
128.82
128.31
127.08
123.33
123.20
121.71
121.12
116.94
115.84

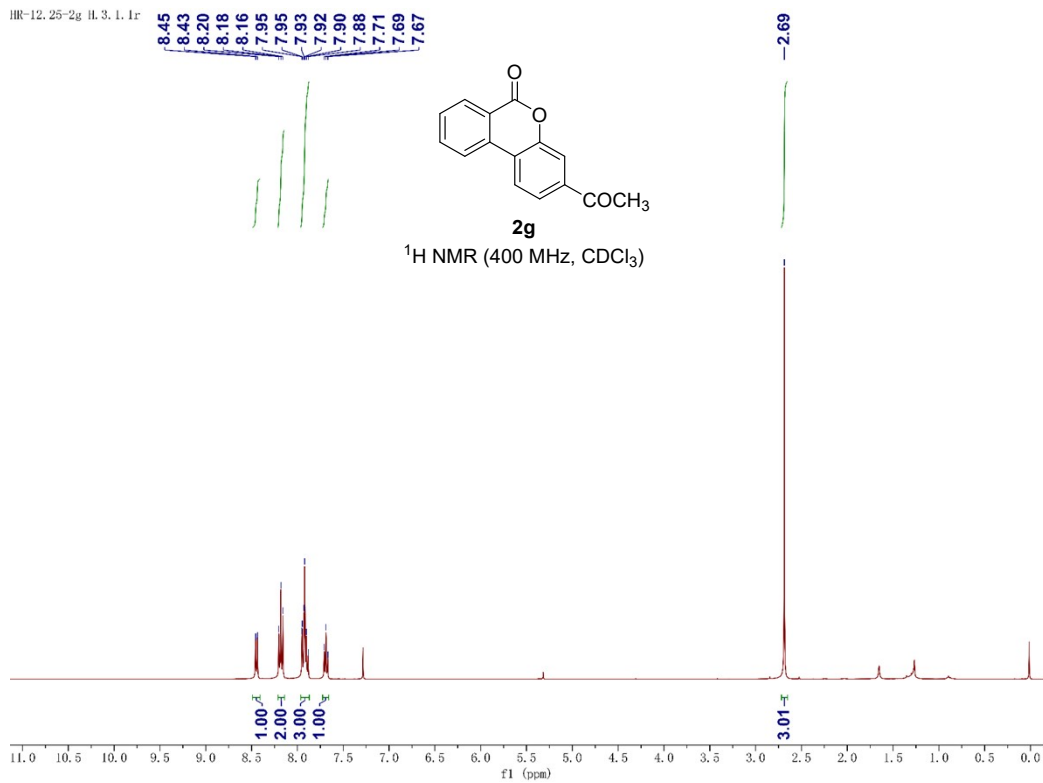


2f

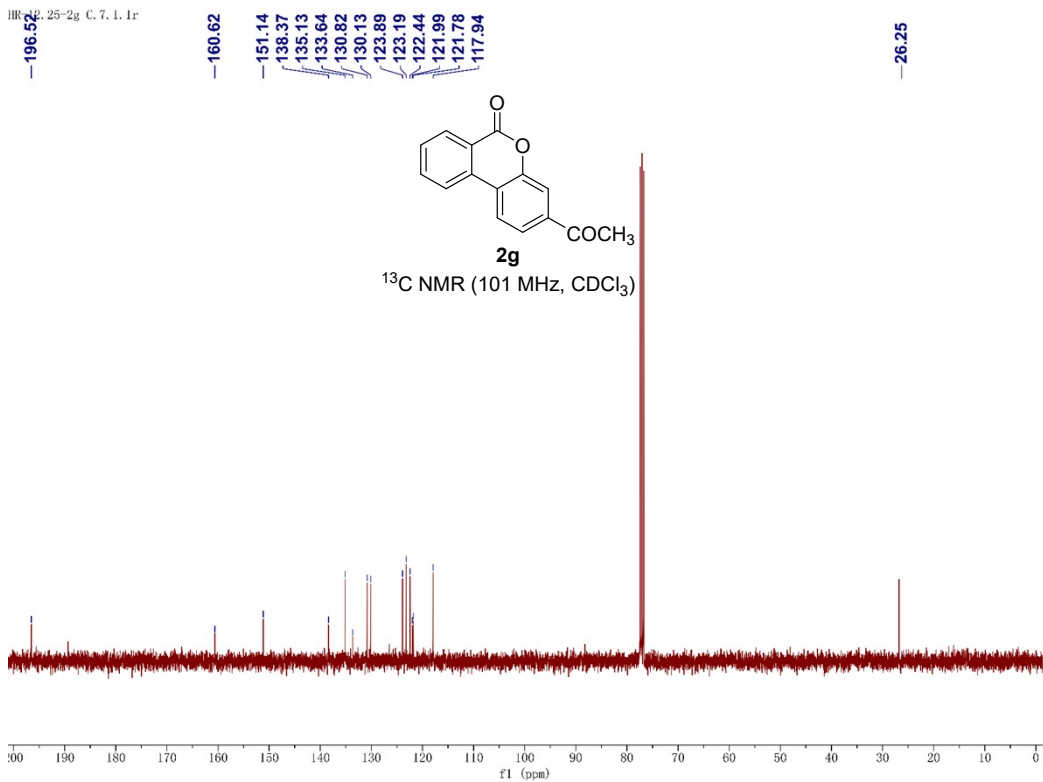
¹³C NMR (126 MHz, CDCl₃)

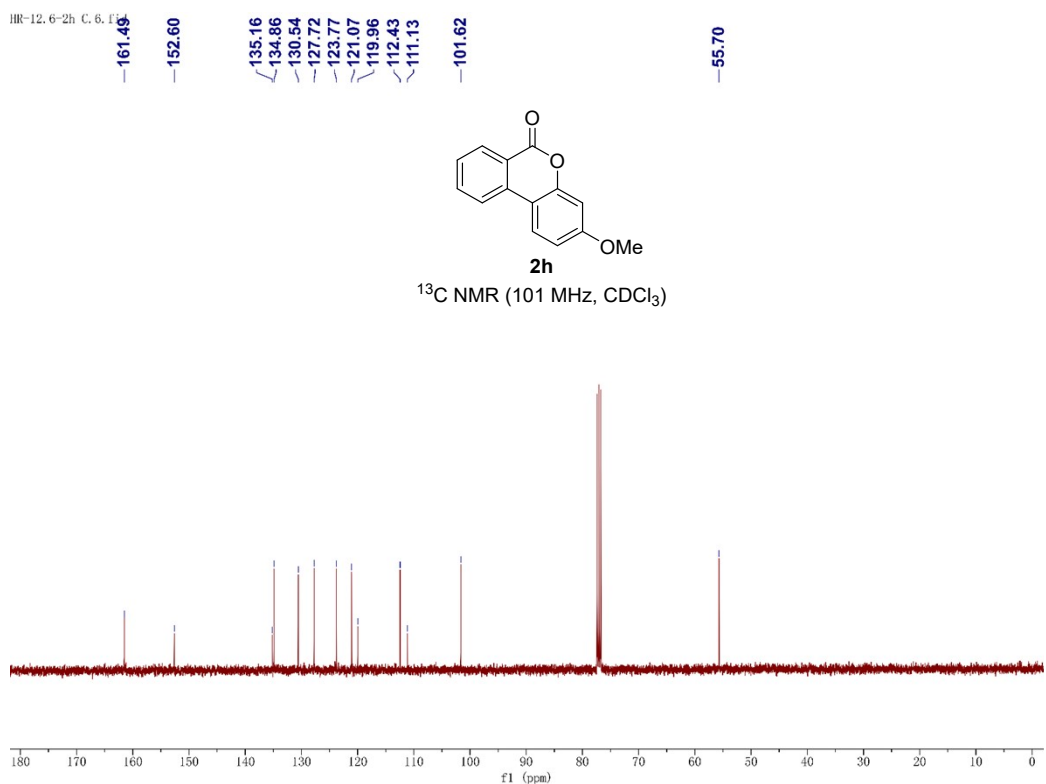
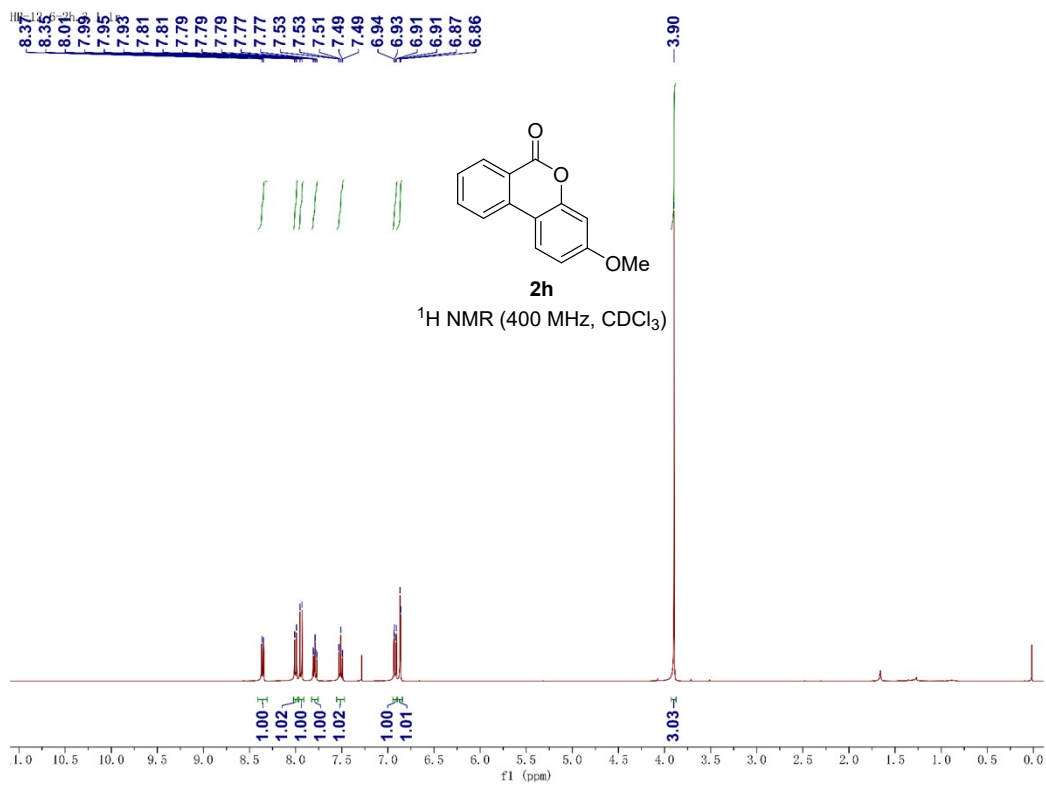


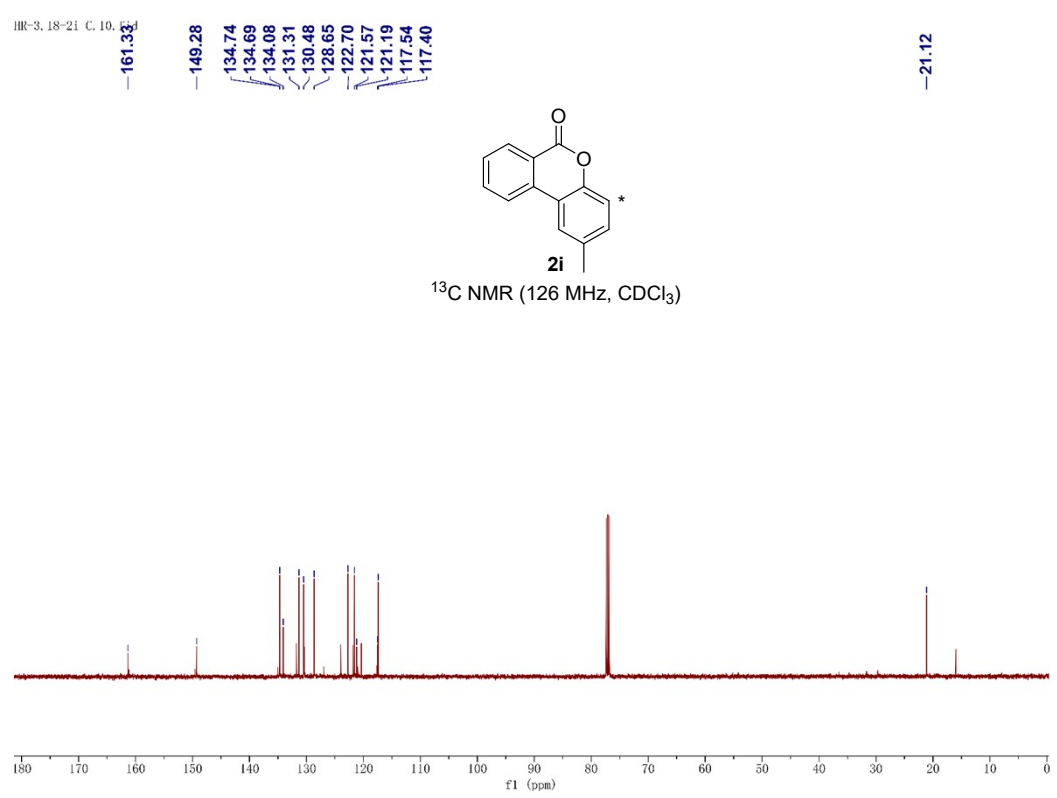
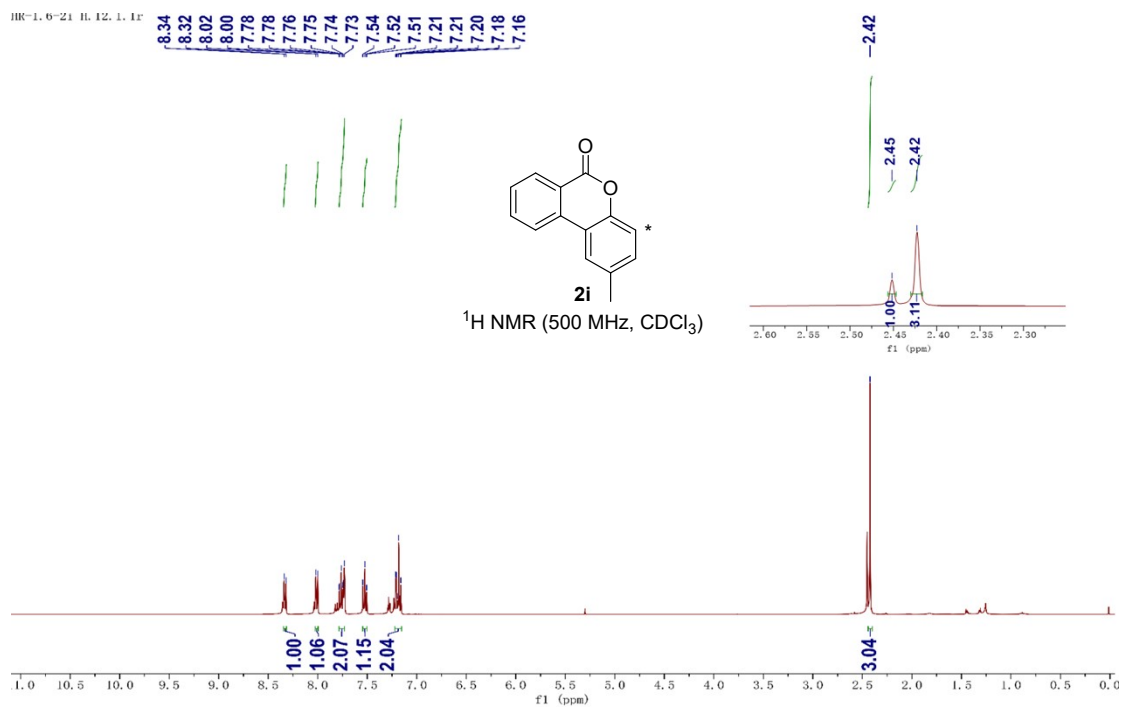
HR-12-25-2g H. 3. 1. 1r



HR-12-25-2g C. 7. 1. 1r

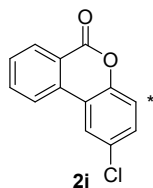
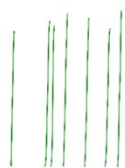




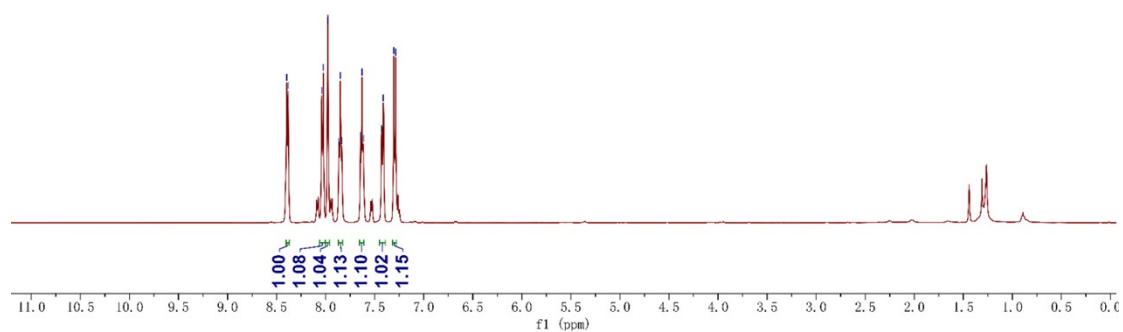
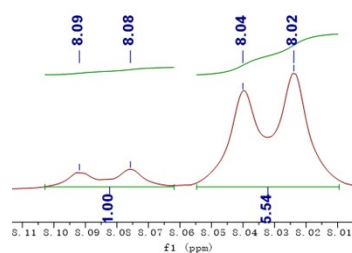


HR-3, 18-2J H, 10, 1, 1F

8.40
8.38
8.04
8.02
7.98
7.87
7.85
7.84
7.65
7.63
7.43
7.41
7.31
7.29

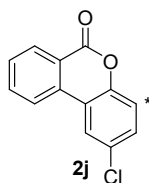


¹H NMR (500 MHz, CDCl₃)

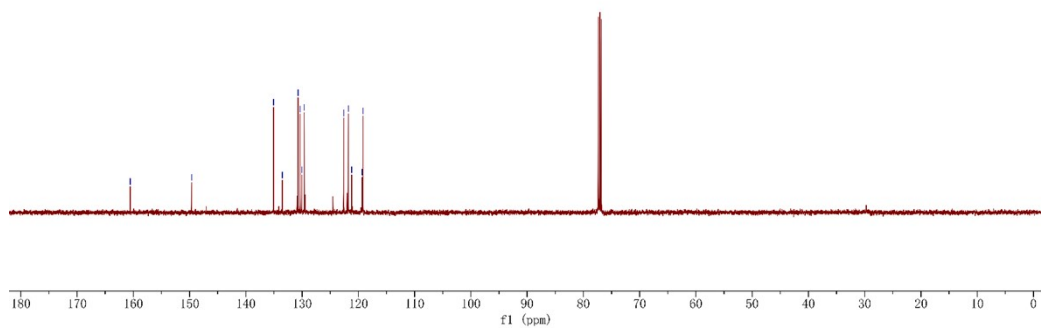


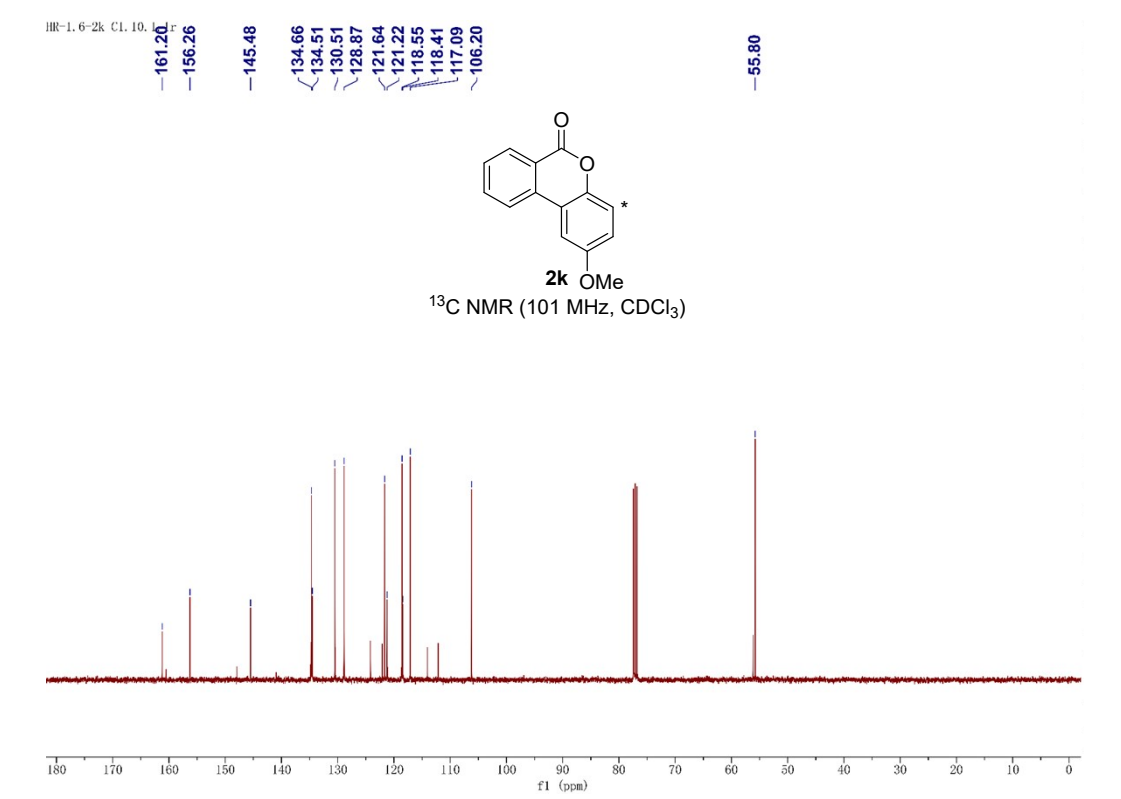
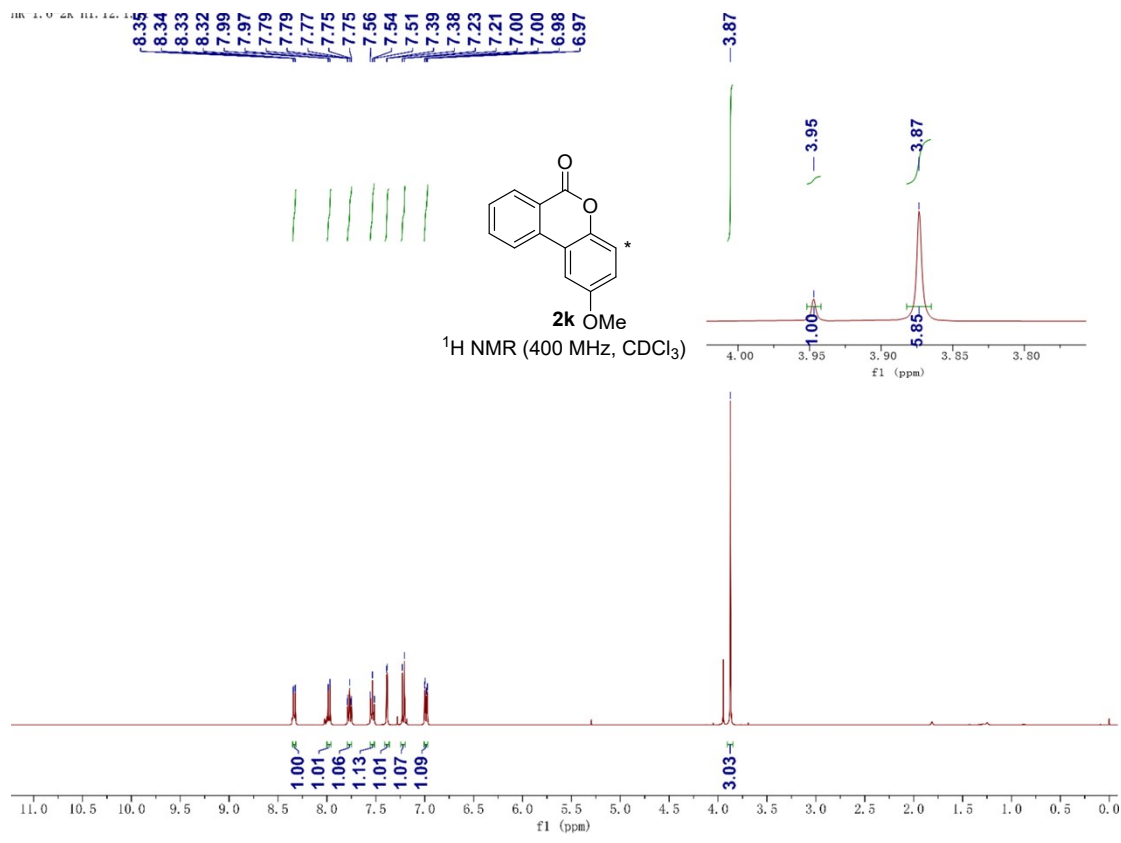
HR-3, 18-2J C, 12, 1F

160.55
149.64
135.07
133.52
130.71
130.36
130.06
129.62
122.59
121.77
121.20
119.33
119.17

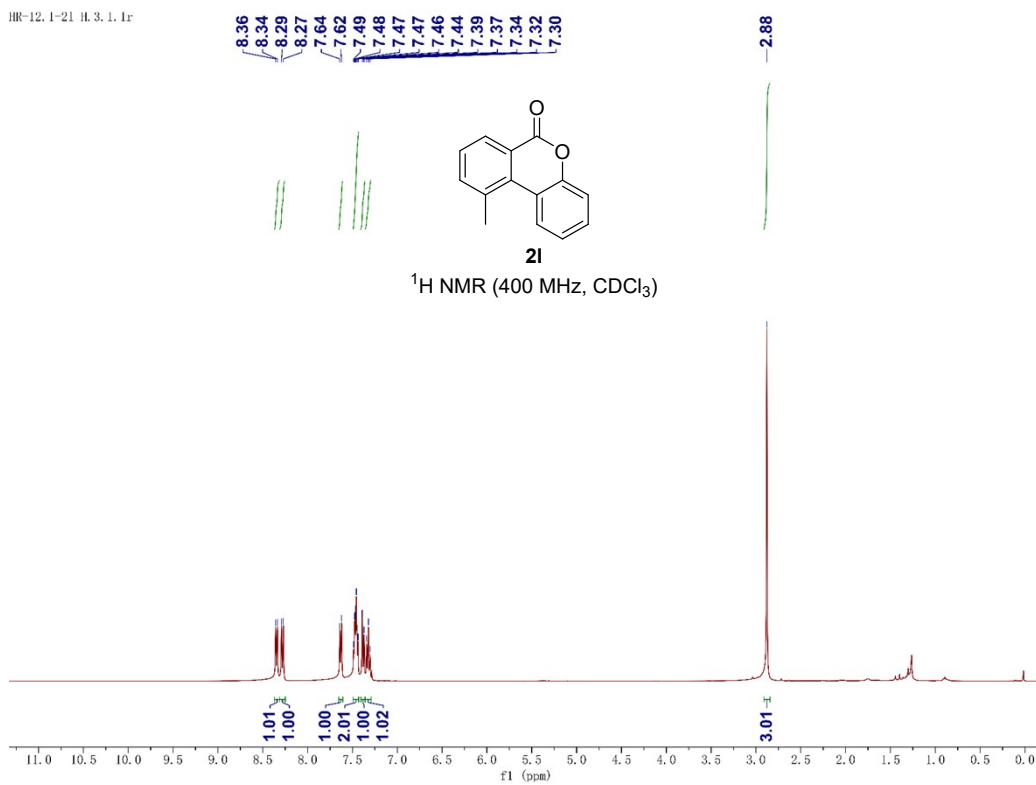


¹³C NMR (126 MHz, CDCl₃)

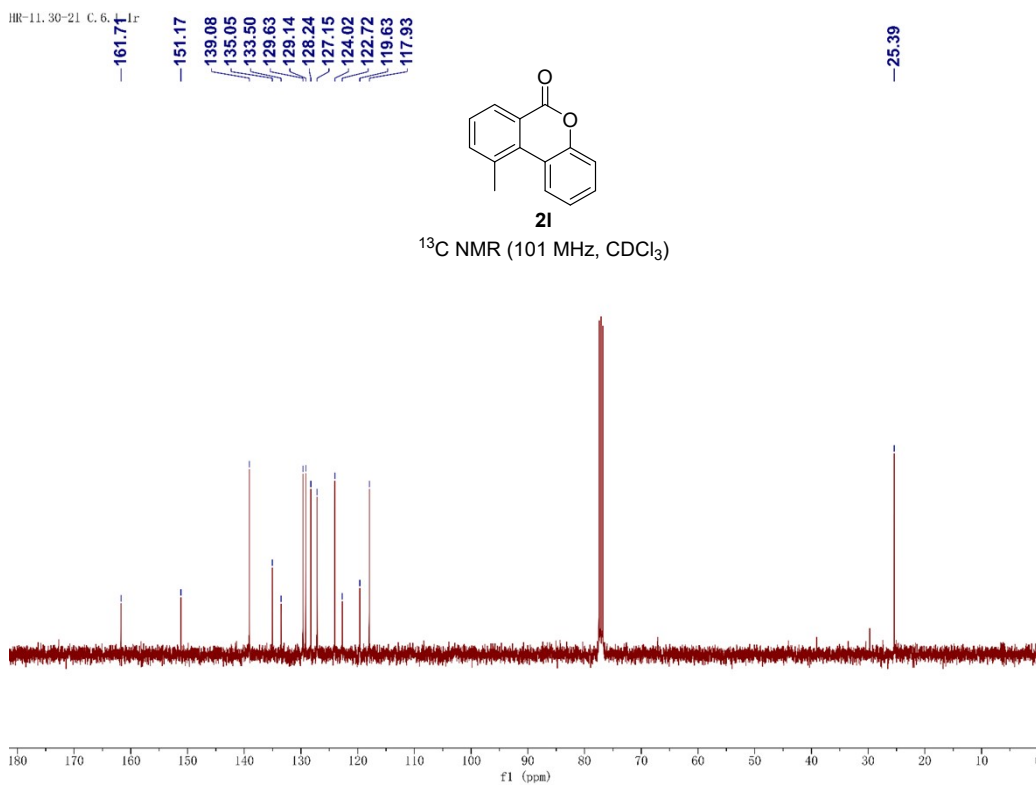




HR-12.1-21 H.3.1.1r

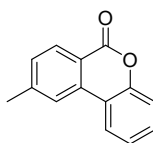


HR-11.30-21 C.6.1r



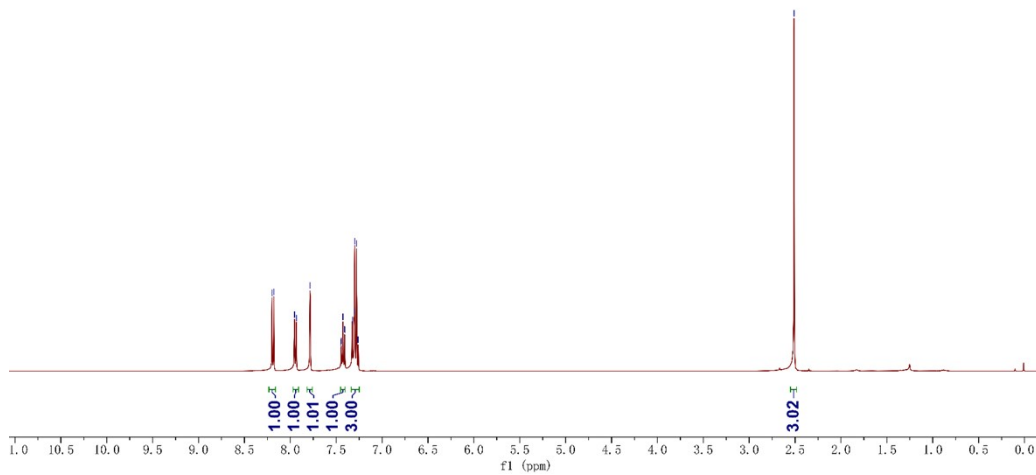
HR-12.7-2m.4.1.1r

8.20
8.18
7.95
7.93
7.79
7.45
7.44
7.43
7.41
7.41
7.32
7.30
7.30
7.28
7.26



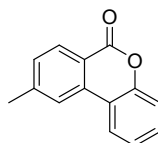
2m

¹H NMR (400 MHz, CDCl₃)



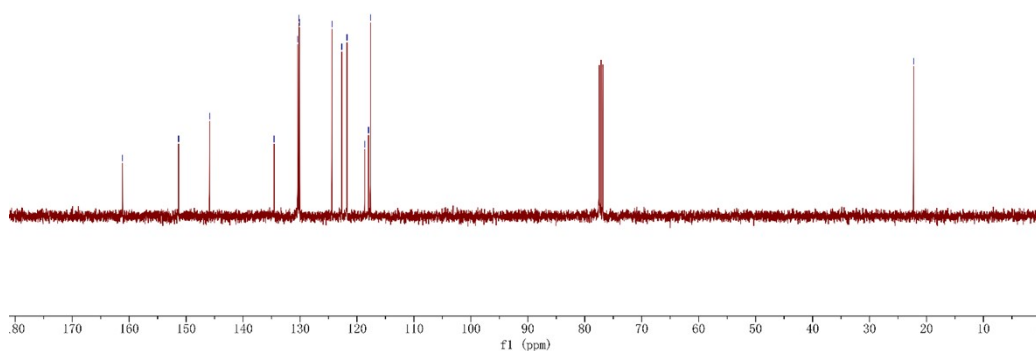
HR-12.7-2m C.12.1r

161.17
151.32
145.87
134.56
130.38
130.21
130.07
124.38
122.66
121.75
118.63
117.96
117.60
22.26

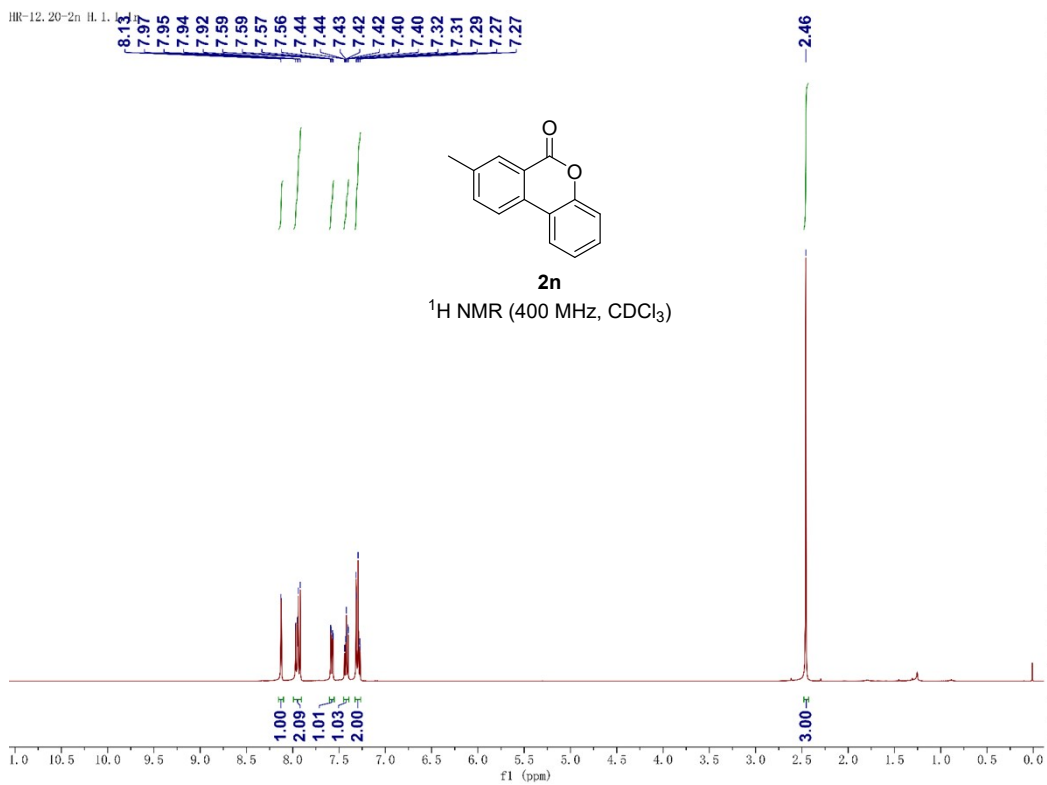


2m

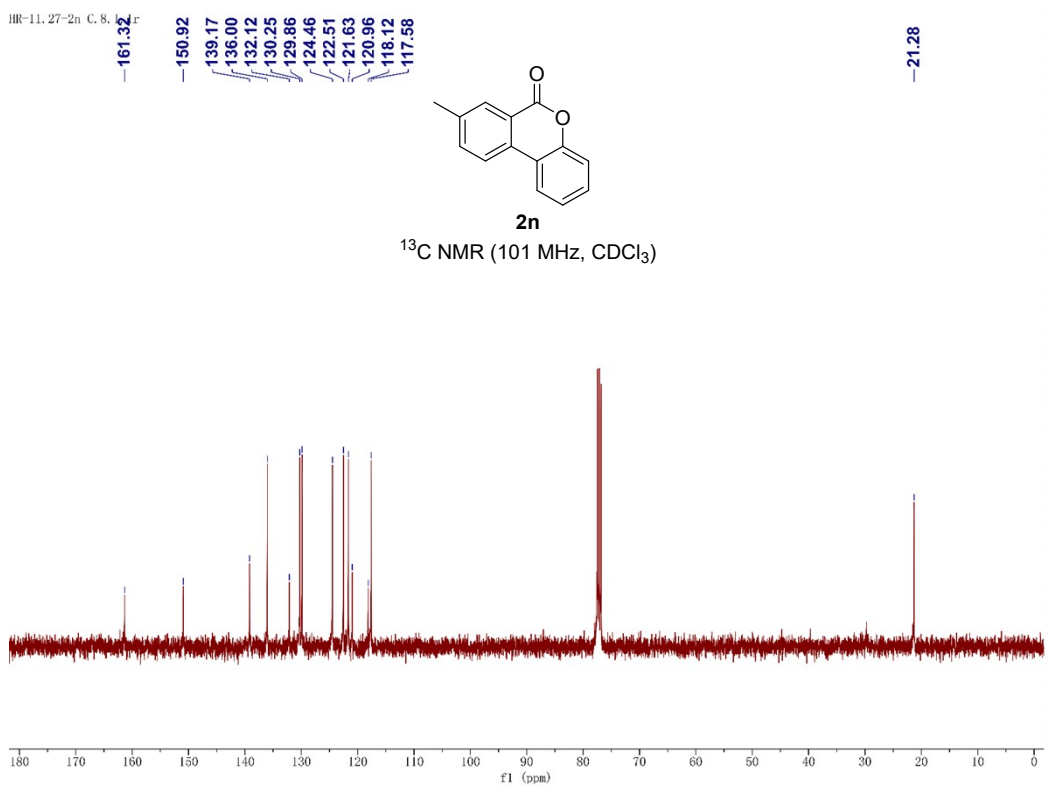
¹³C NMR (101 MHz, CDCl₃)



HR-12.20-2n H.1.

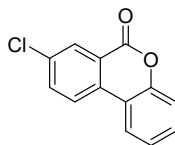
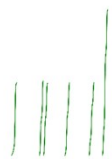


HR-11.27-2n C.8.



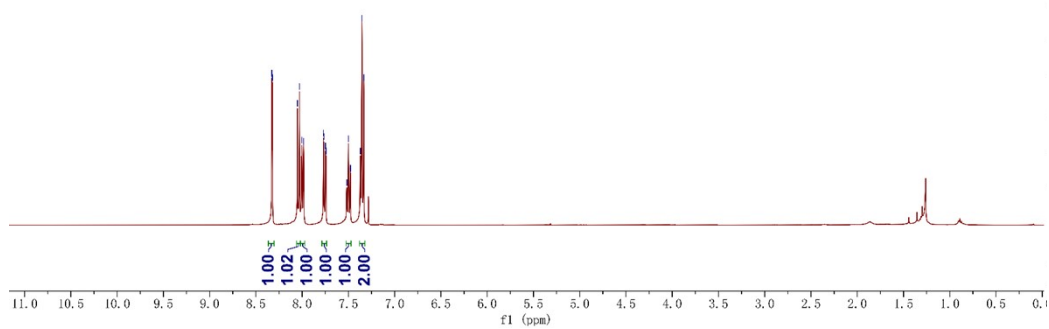
HR-11-28-2o H. 6. 1. Ir

8.33
8.32
8.05
8.03
8.00
7.99
7.77
7.76
7.75
7.74
7.52
7.50
7.48
7.37
7.35
7.33



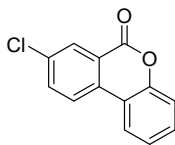
2o

¹H NMR (400 MHz, CDCl₃)



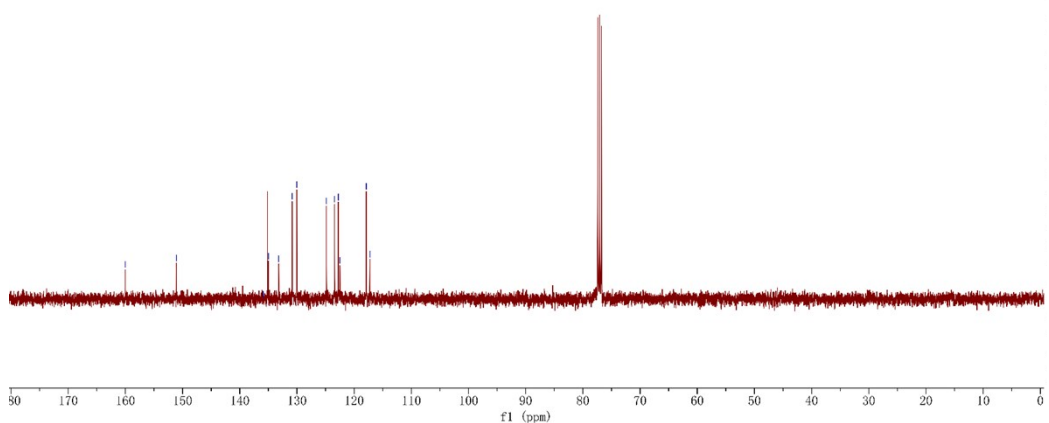
HR-11-24-2o C. 6. 1. Ir

160.03
151.08
135.93
134.98
133.19
130.85
130.03
124.85
123.42
122.76
122.49
117.87
117.24

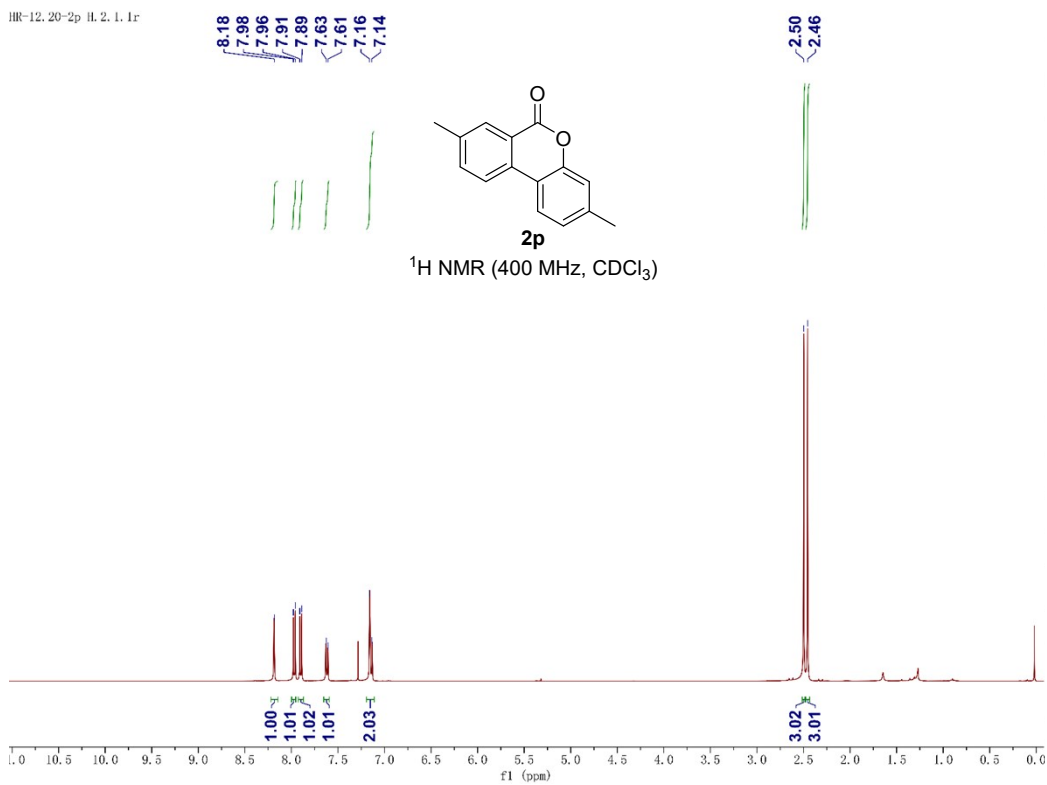


2o

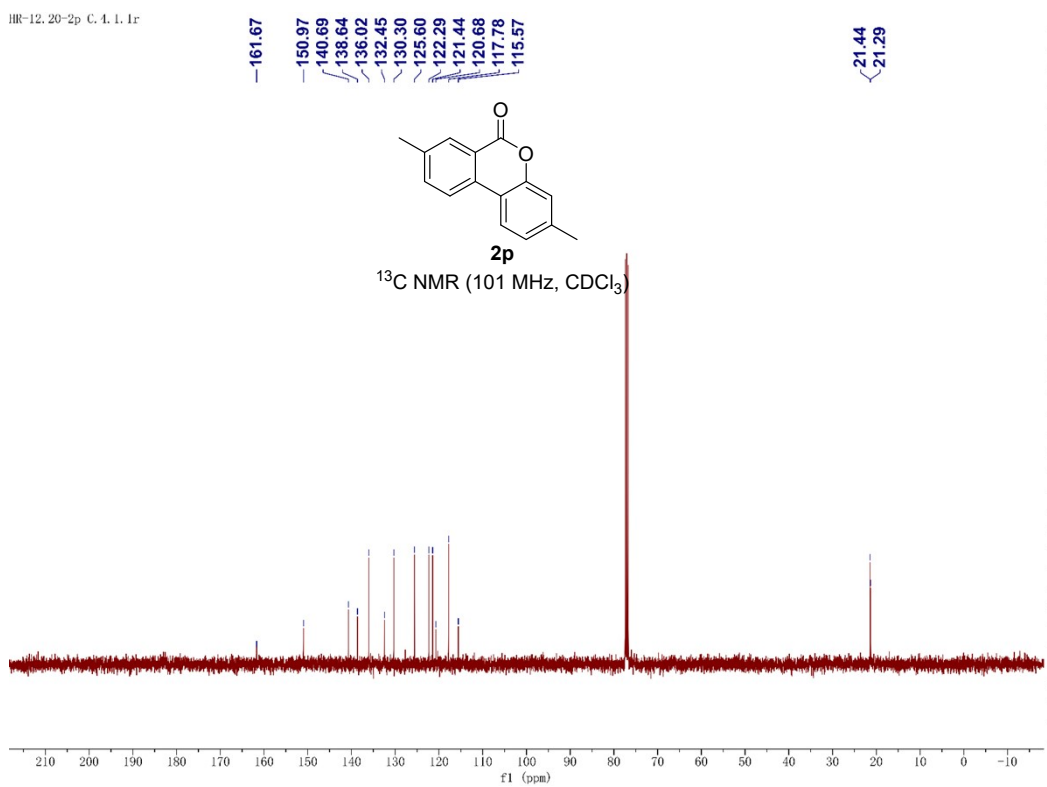
¹³C NMR (101 MHz, CDCl₃)



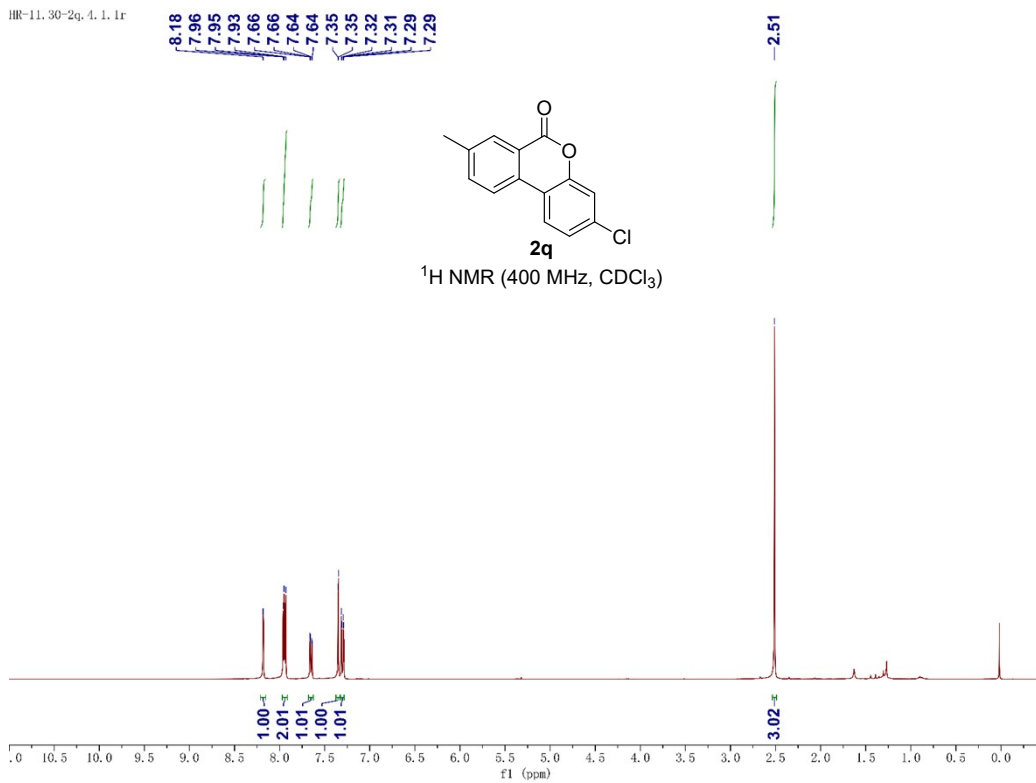
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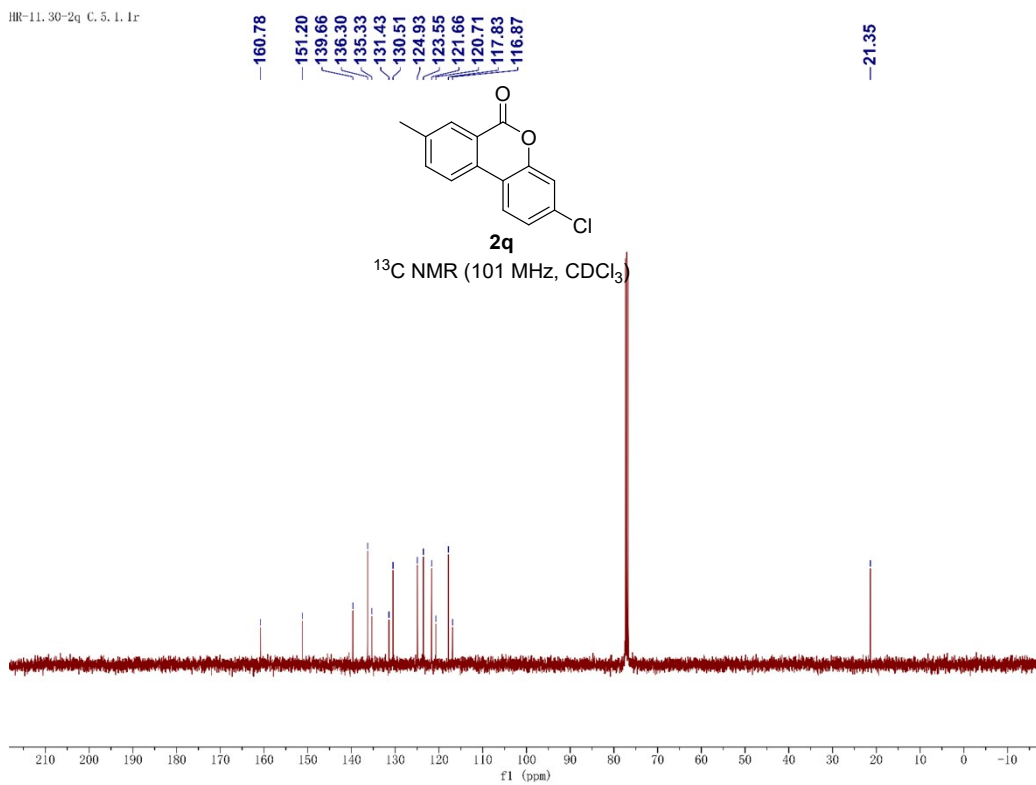
HR-12.20-2p C.4.1.1r



HR-11.30-2q, 4.1.1r

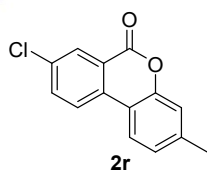


HR-11.30-2q C.5.1.r



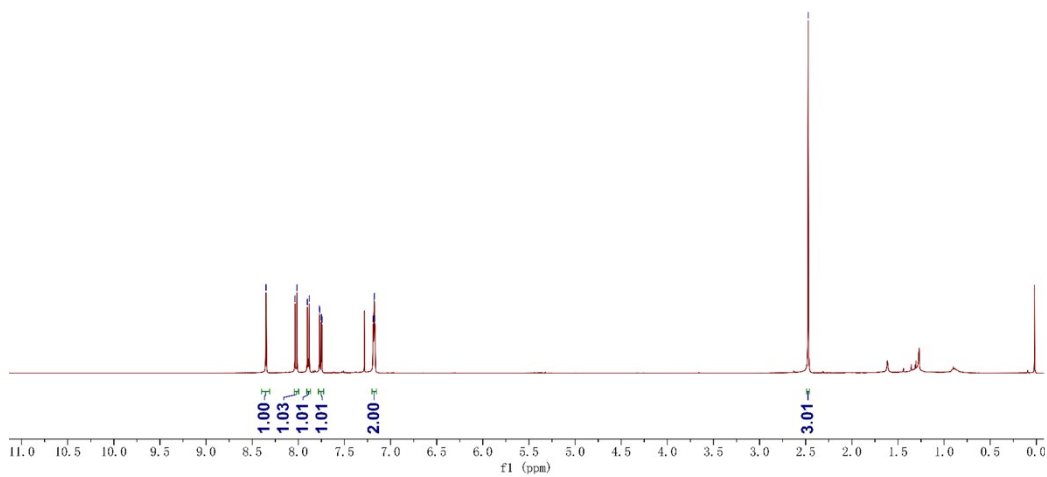
HR-12.18-2r. 8.1.1r

8.35
8.35
8.04
8.01
7.90
7.88
7.77
7.77
7.75
7.74
7.19
7.18
7.18
7.17



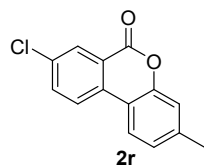
¹H NMR (400 MHz, CDCl₃)

2.47



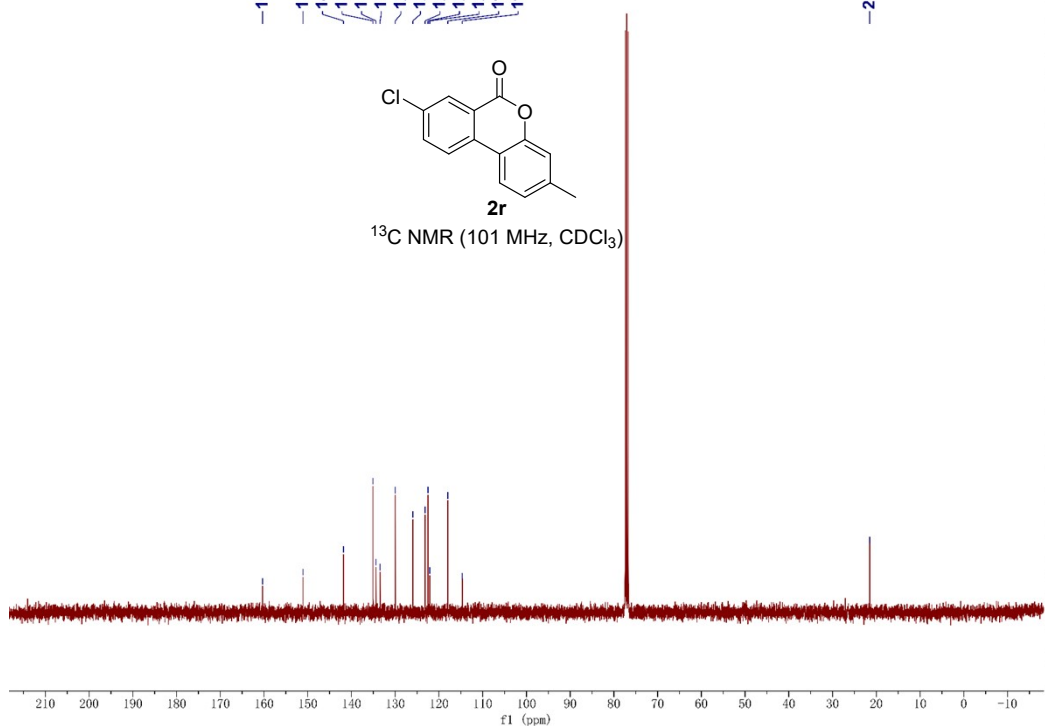
HR-12.20-2r C.1.1.1r

160.32
151.09
141.81
135.08
134.39
133.45
129.99
125.97
122.50
123.18
122.11
117.97
114.66



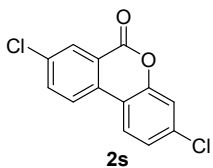
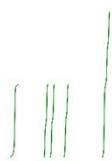
¹³C NMR (101 MHz, CDCl₃)

21.52

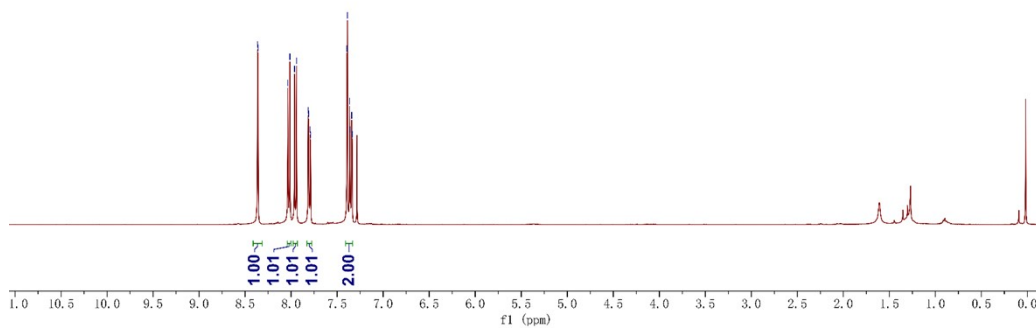


HR-12.5-2s.6.1.1r

8.36
8.36
8.04
8.01
7.96
7.94
7.82
7.81
7.79
7.79
7.39
7.39
7.36
7.36
7.34
7.34

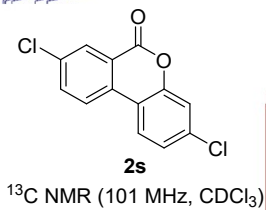


¹H NMR (400 MHz, CDCl₃)

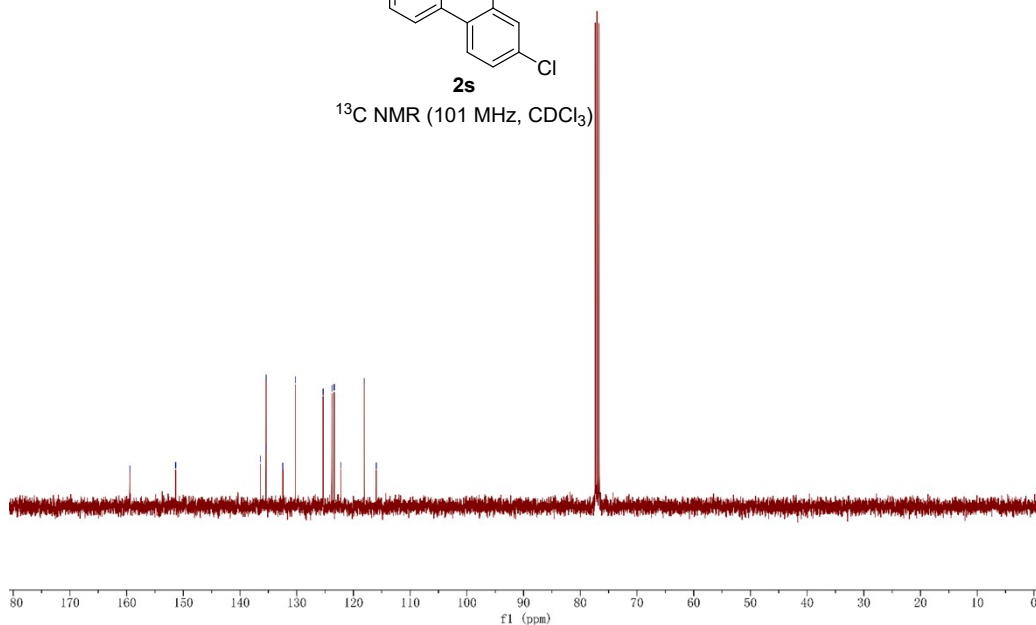


HR-2s C.1.fid

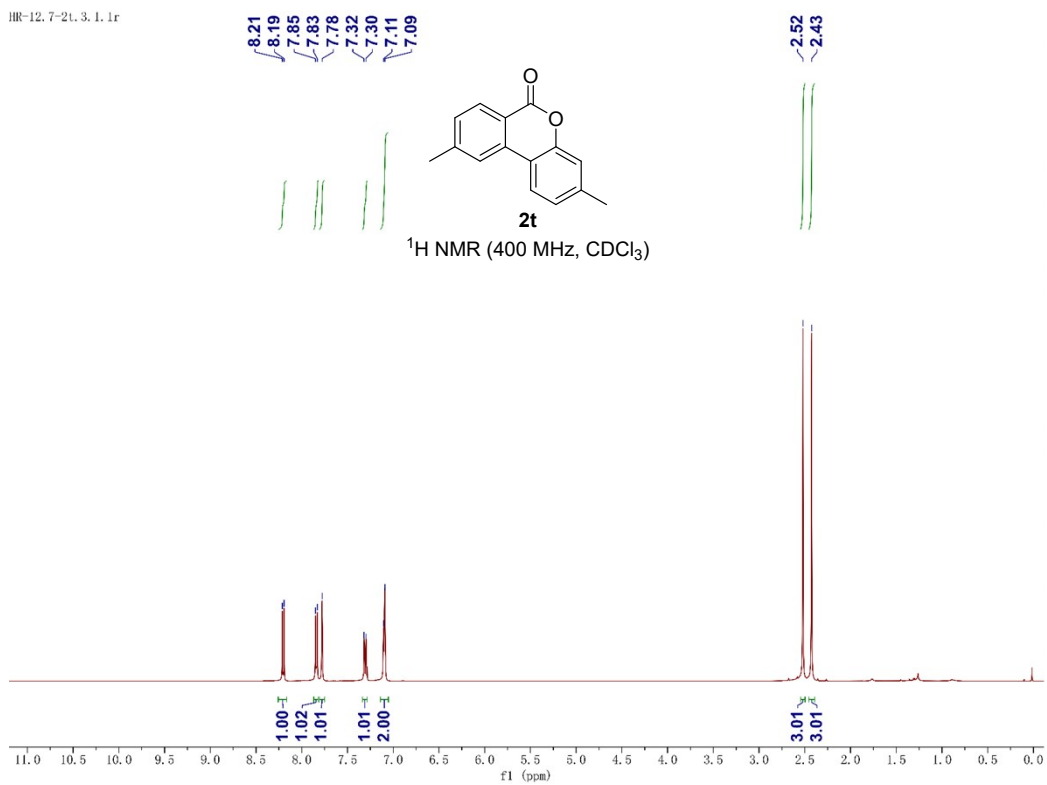
159.43
151.35
136.40
135.39
135.37
132.46
130.23
125.33
123.77
123.39
122.20
118.09
115.98



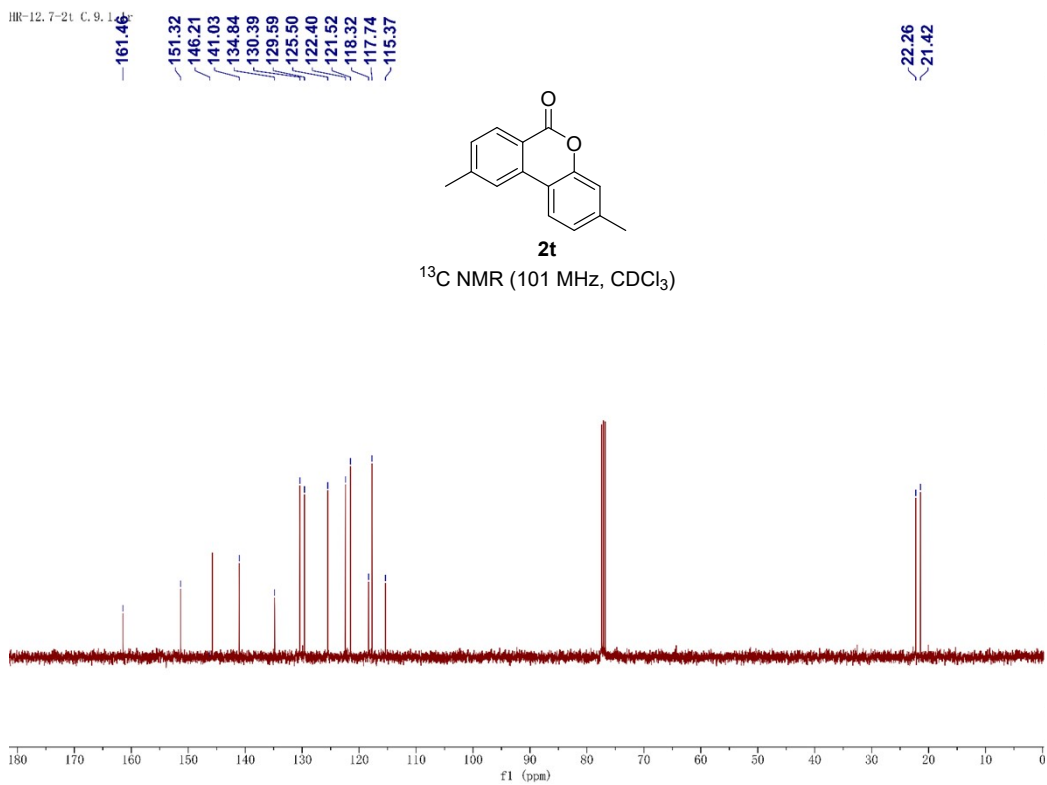
¹³C NMR (101 MHz, CDCl₃)



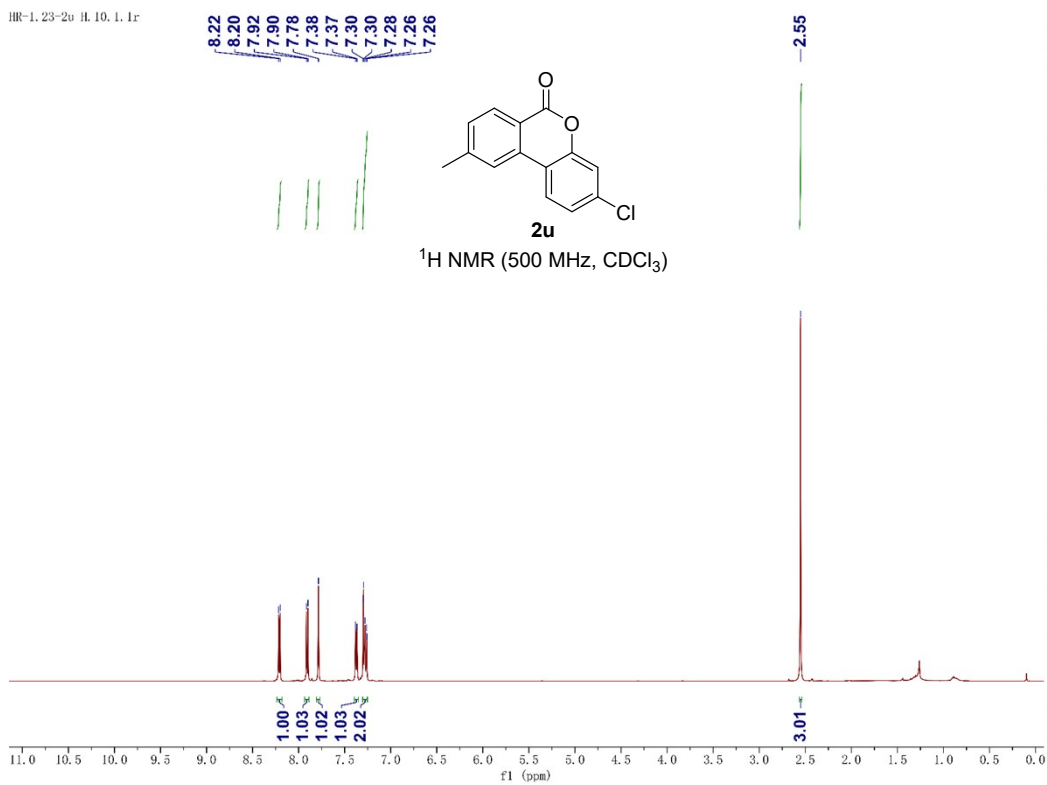
HR-12.7-21.3.1.1r



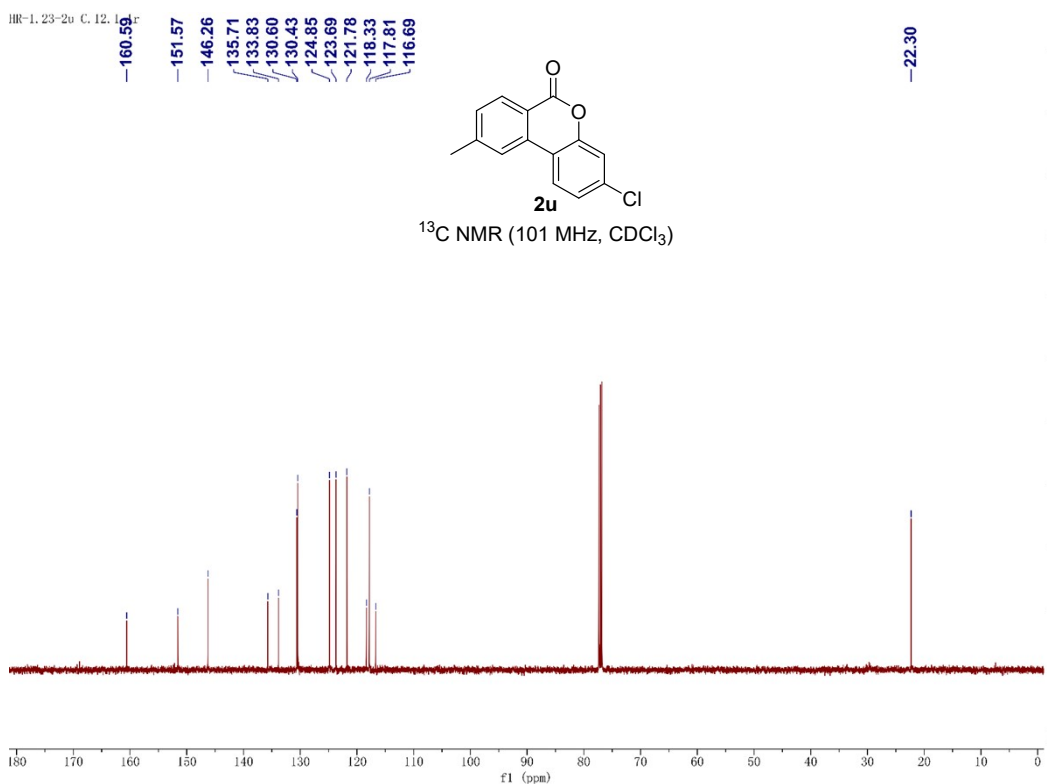
HR-12.7-21.3.9.1r



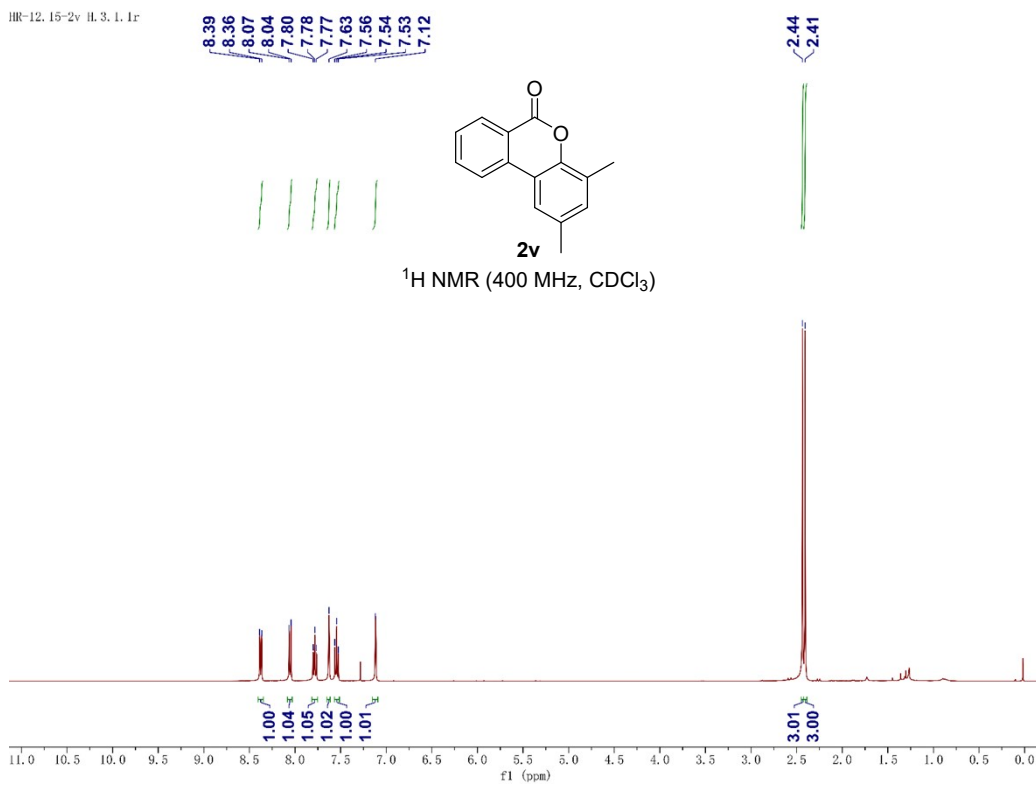
HR-1.23-2u H. 10. 1. Ir



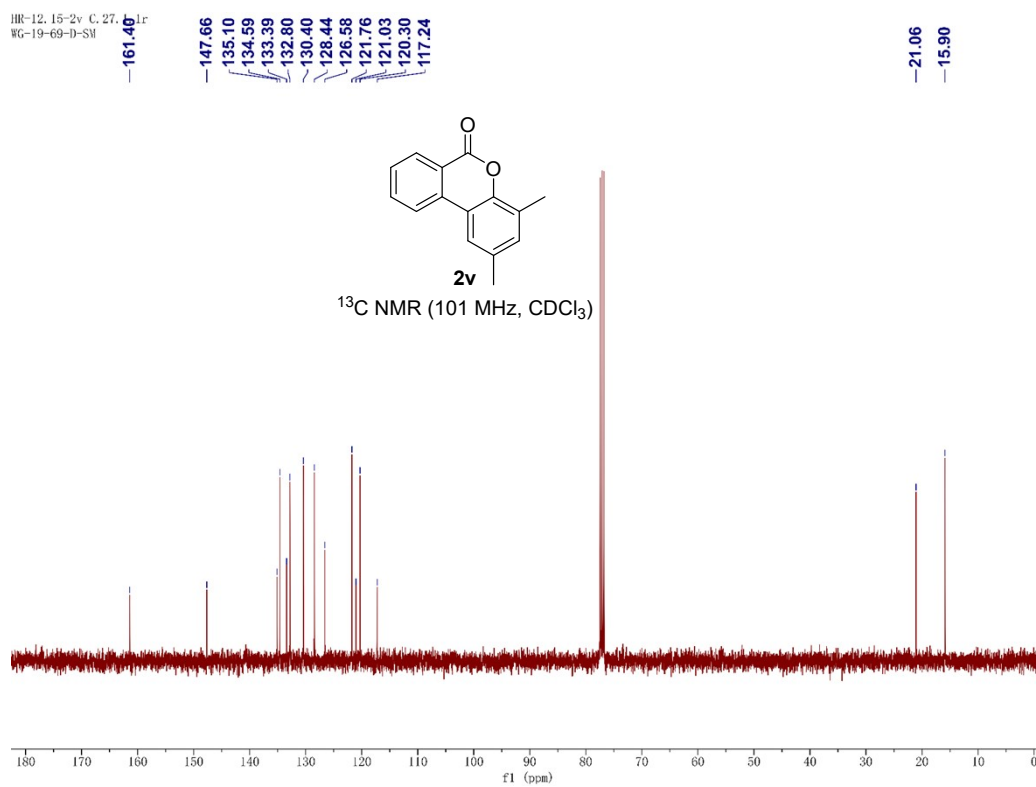
HR-1.23-2u C. 12.



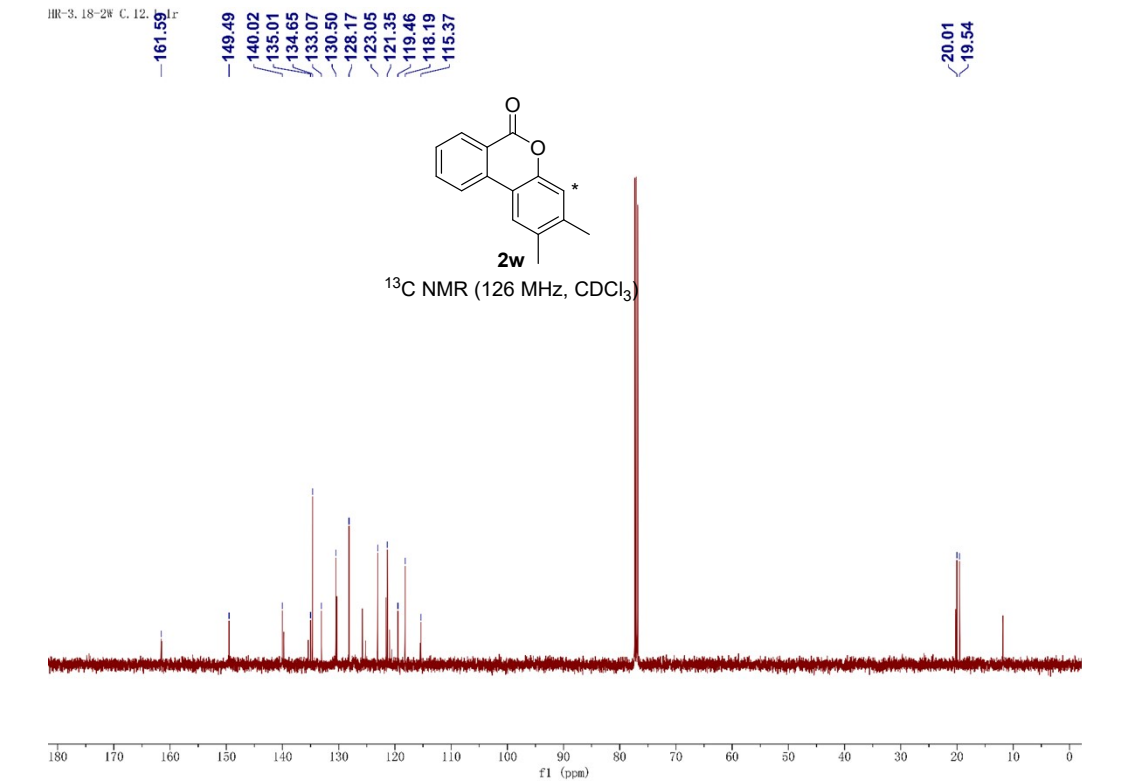
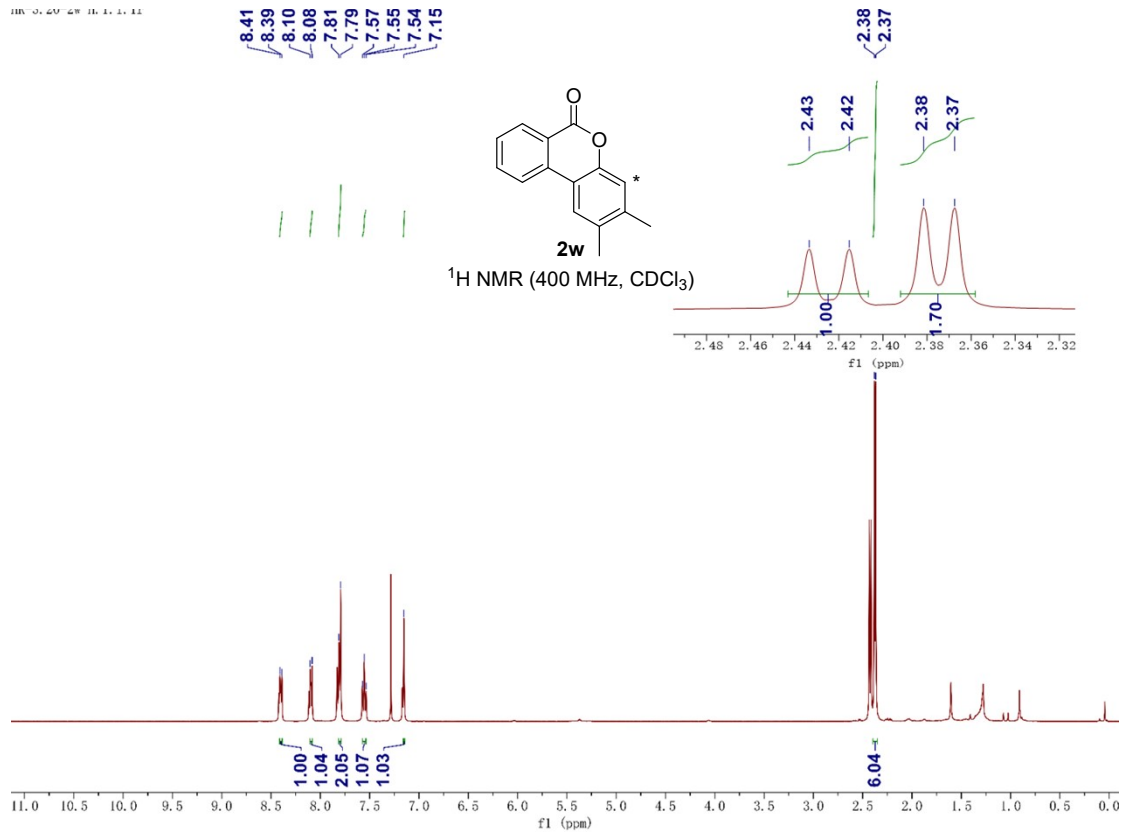
HR-12, 15-2v H.3.1.1r



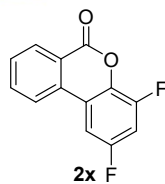
HR-12, 15-2v C.27
WG-19-69-D-SM



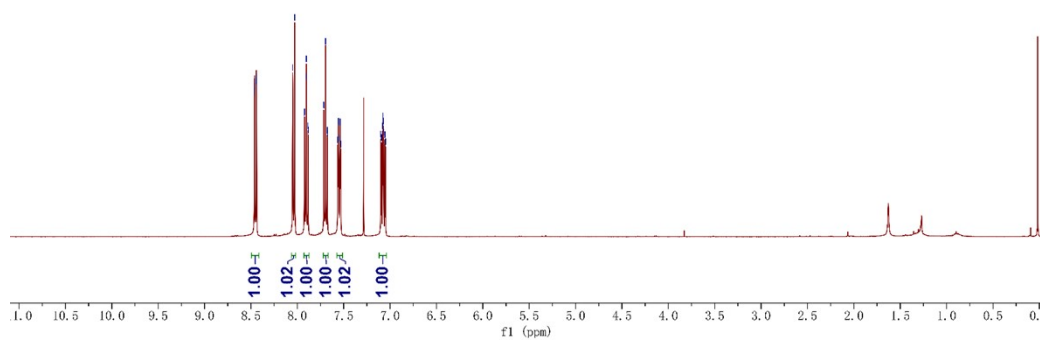
HR-3-20-2W H. 1. 1. 11



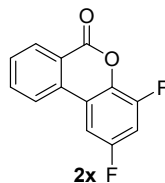
HR-15
8.46
8.46
8.44
8.44
8.05
8.03
7.92
7.91
7.90
7.89
7.88
7.72
7.71
7.70
7.56
7.54
7.10
7.09
7.08
7.07
7.07
7.06
7.05



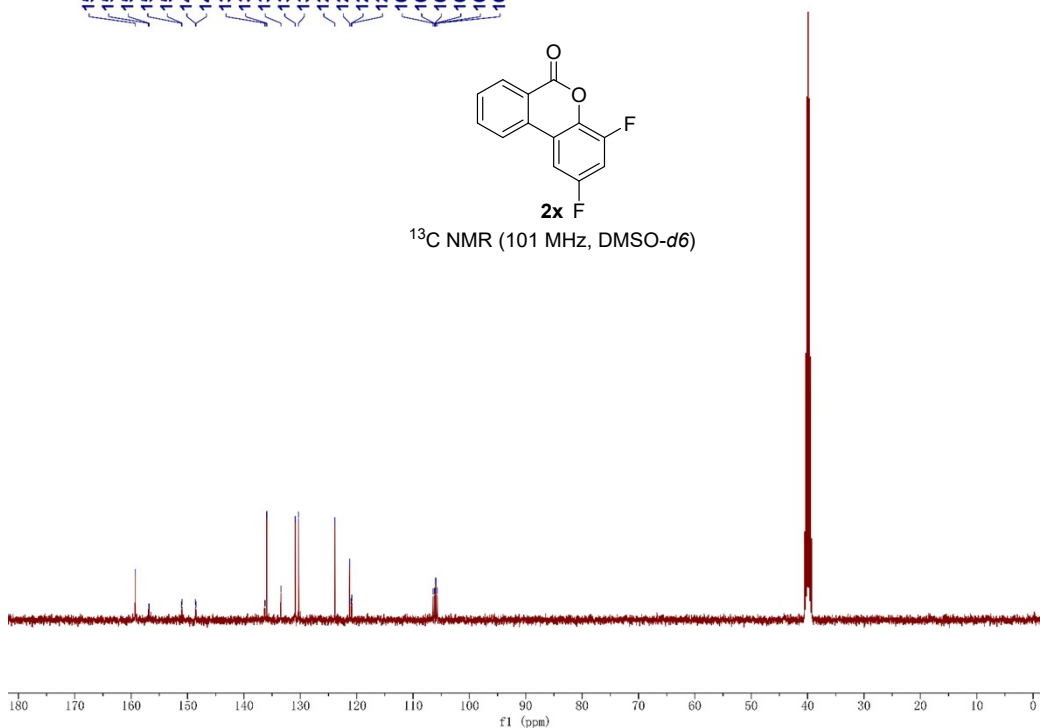
¹H NMR (400 MHz, CDCl₃)

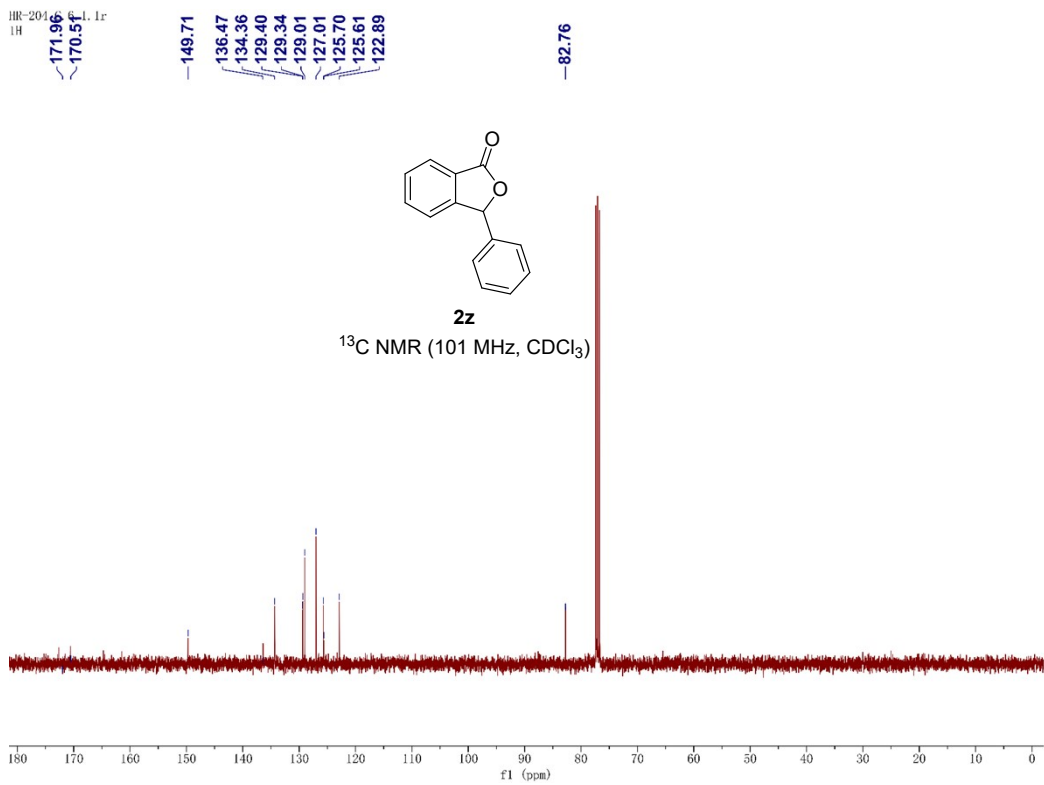
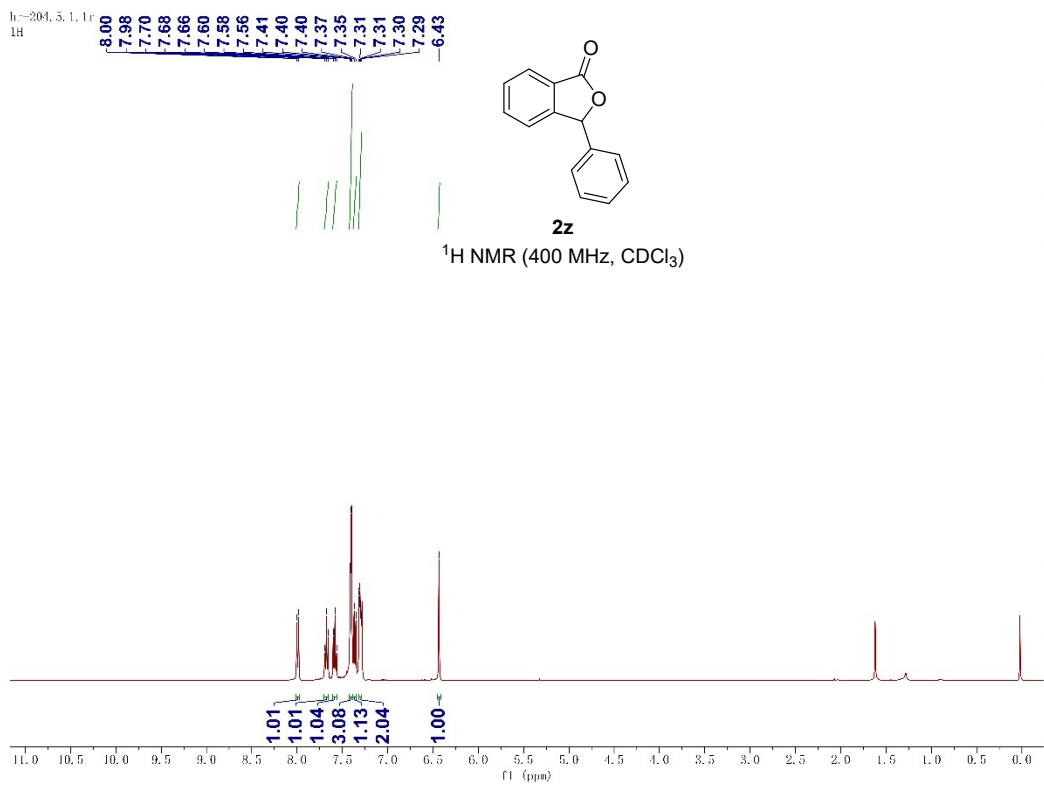


HR-12, 15-2x
WG-19-69-D
159.26
156.98
156.84
151.10
150.96
148.62
148.48
136.25
135.94
133.43
130.90
130.30
123.87
121.24
120.96
120.87
106.45
106.24
106.17
105.96
105.73
105.69



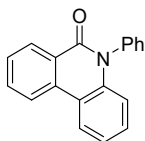
¹³C NMR (101 MHz, DMSO-d₆)





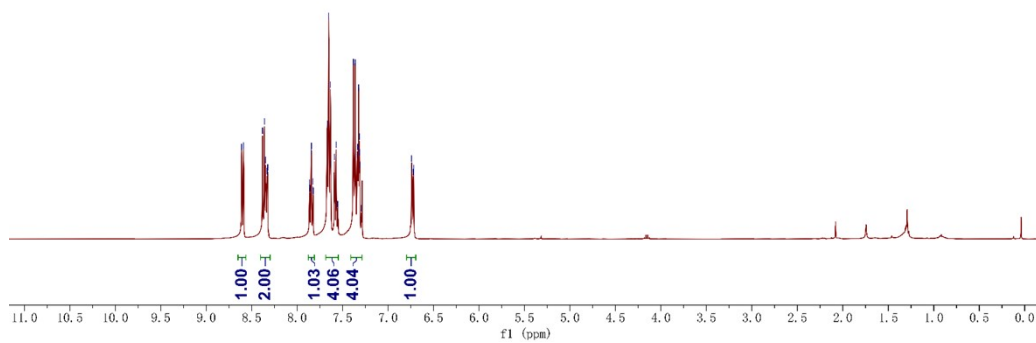
HR-12.18-4a.10.1.1r

8.61
8.59
8.38
8.36
8.35
8.33
8.32
7.86
7.85
7.84
7.83
7.82
7.67
7.65
7.63
7.59
7.57
7.55
7.38
7.36
7.34
7.33
7.32
7.31
7.29
6.74
6.72
6.72



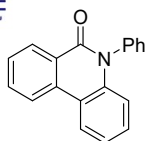
4a

¹H NMR (400 MHz, CDCl₃)



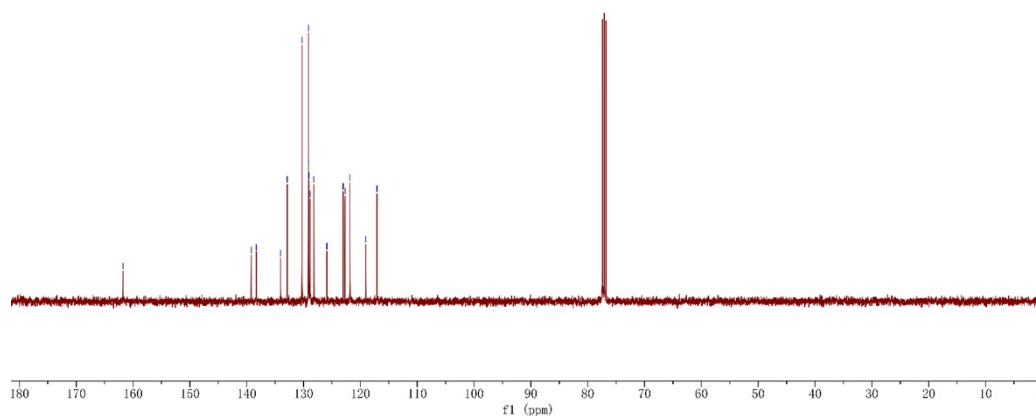
HR-12.20-4a C.2.1r

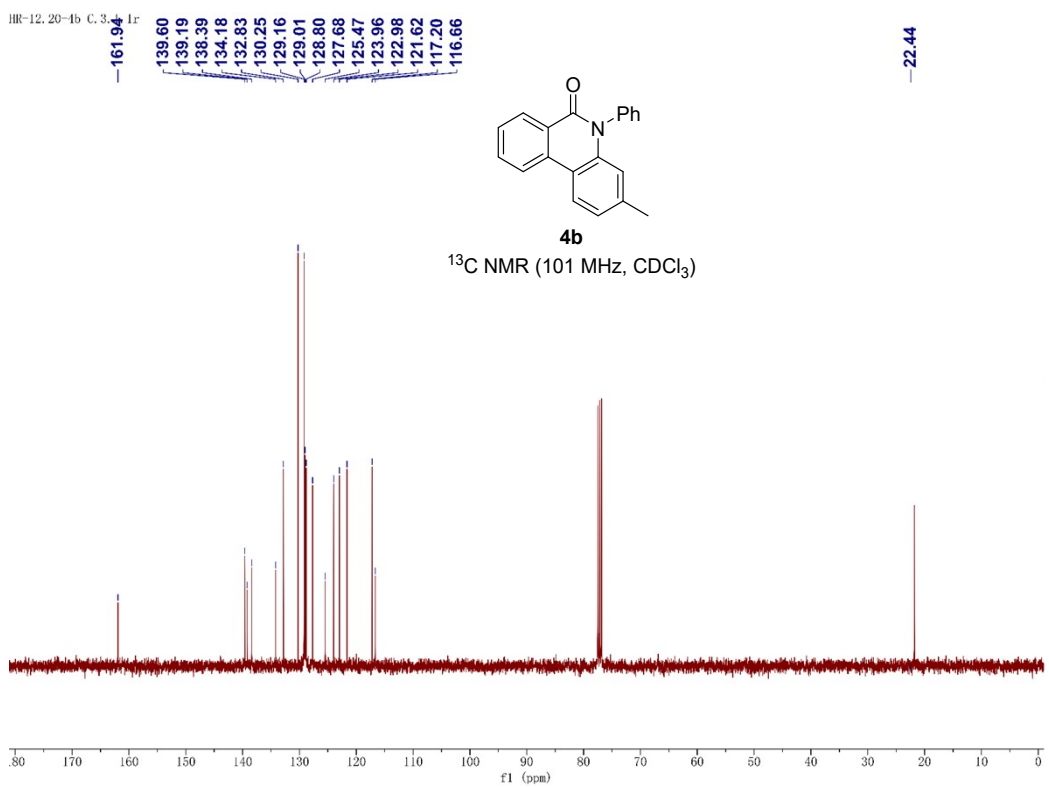
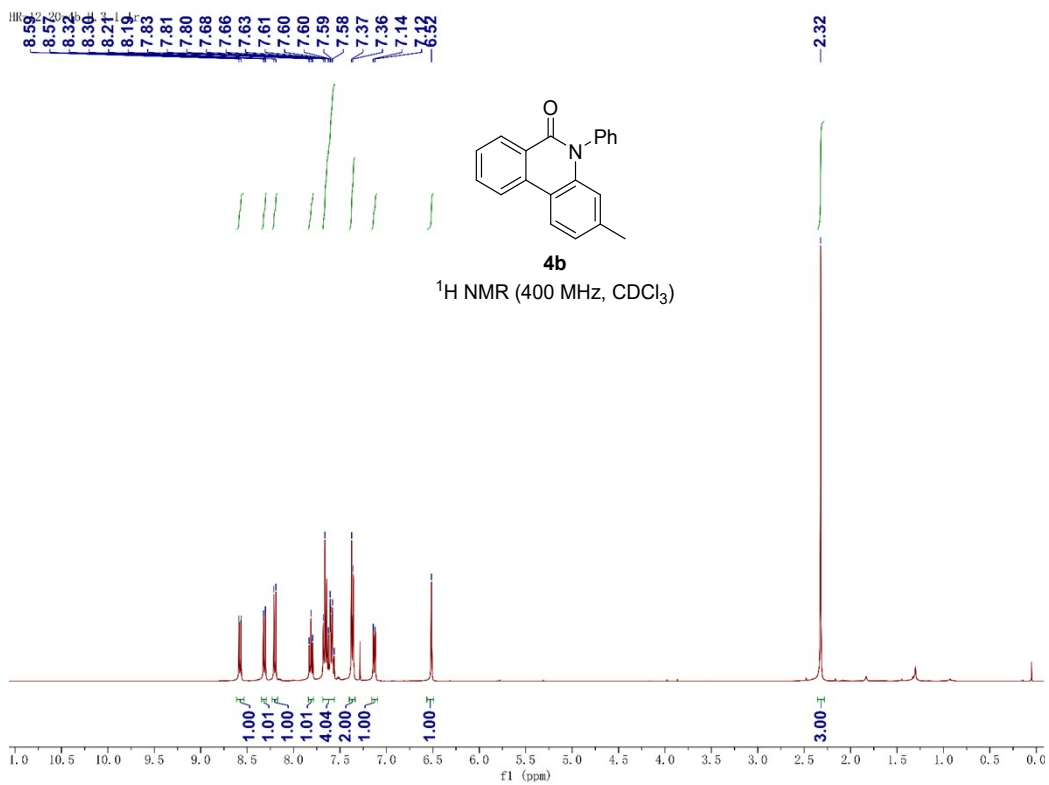
161.76
139.20
138.32
134.04
132.88
130.26
129.14
129.12
129.06
128.84
128.18
125.89
123.03
122.69
121.84
119.06
117.07

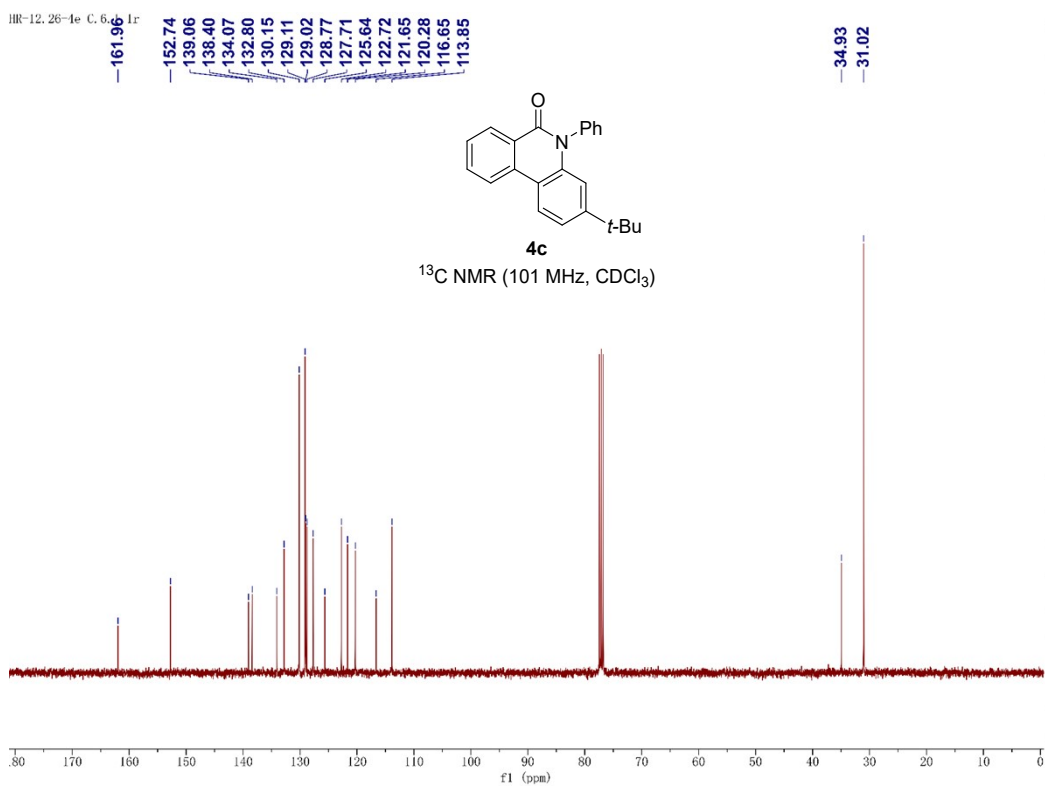
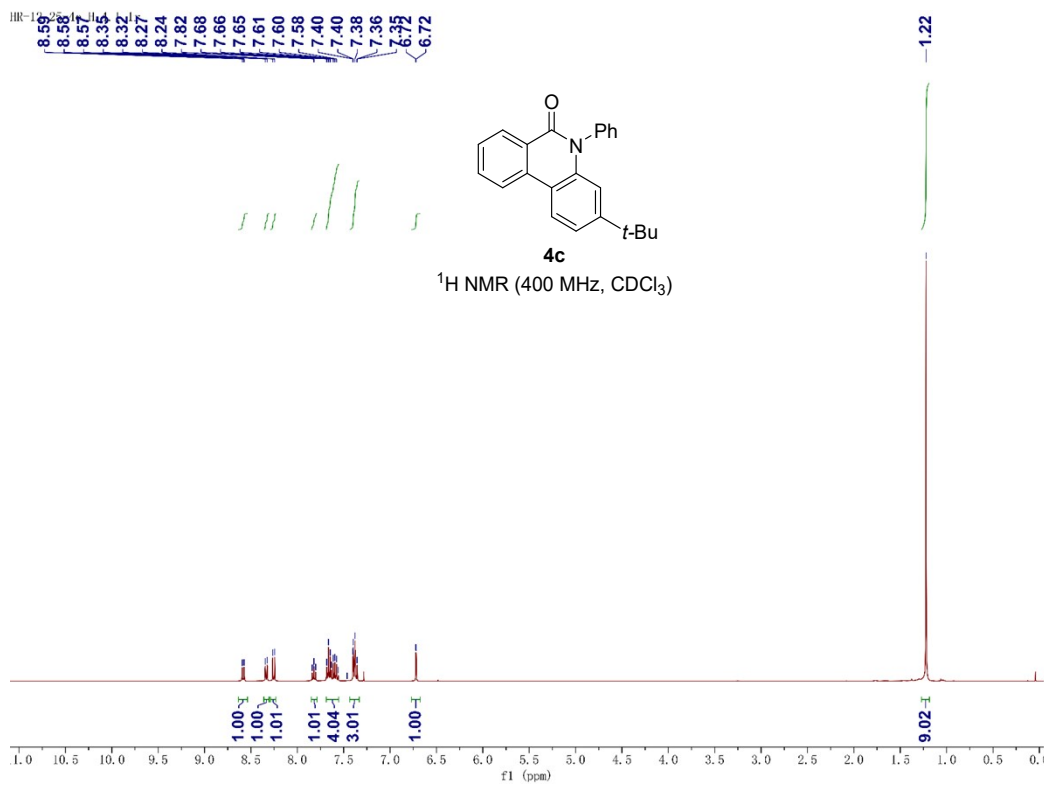


4a

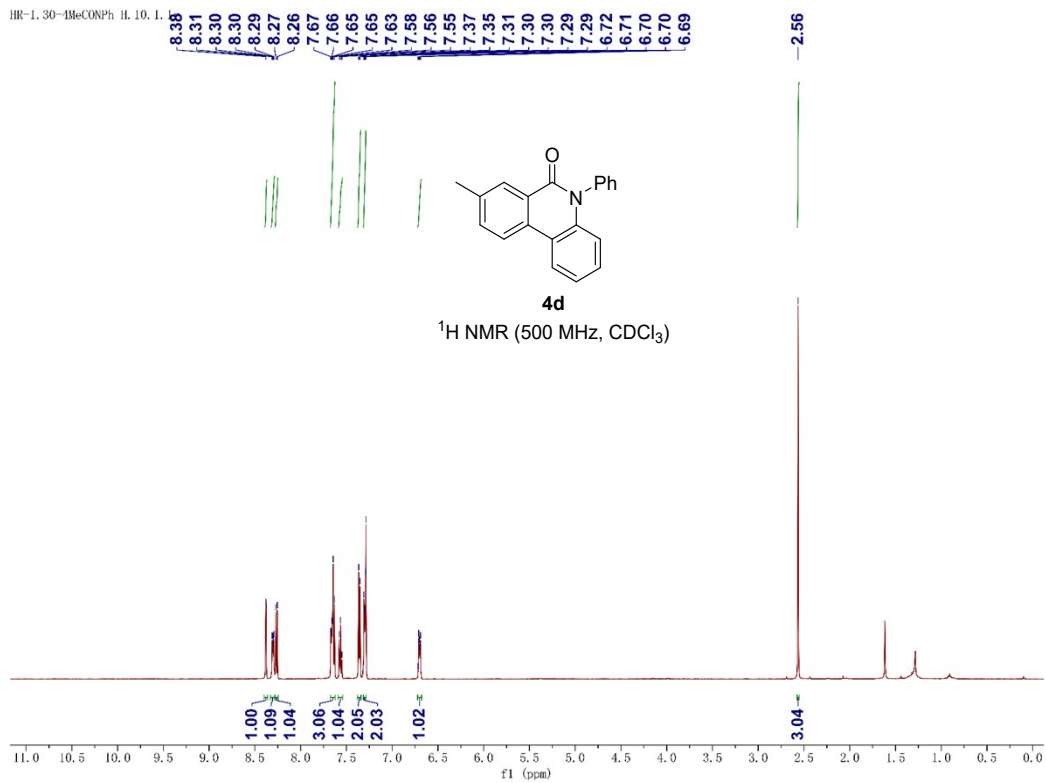
¹³C NMR (101 MHz, CDCl₃)



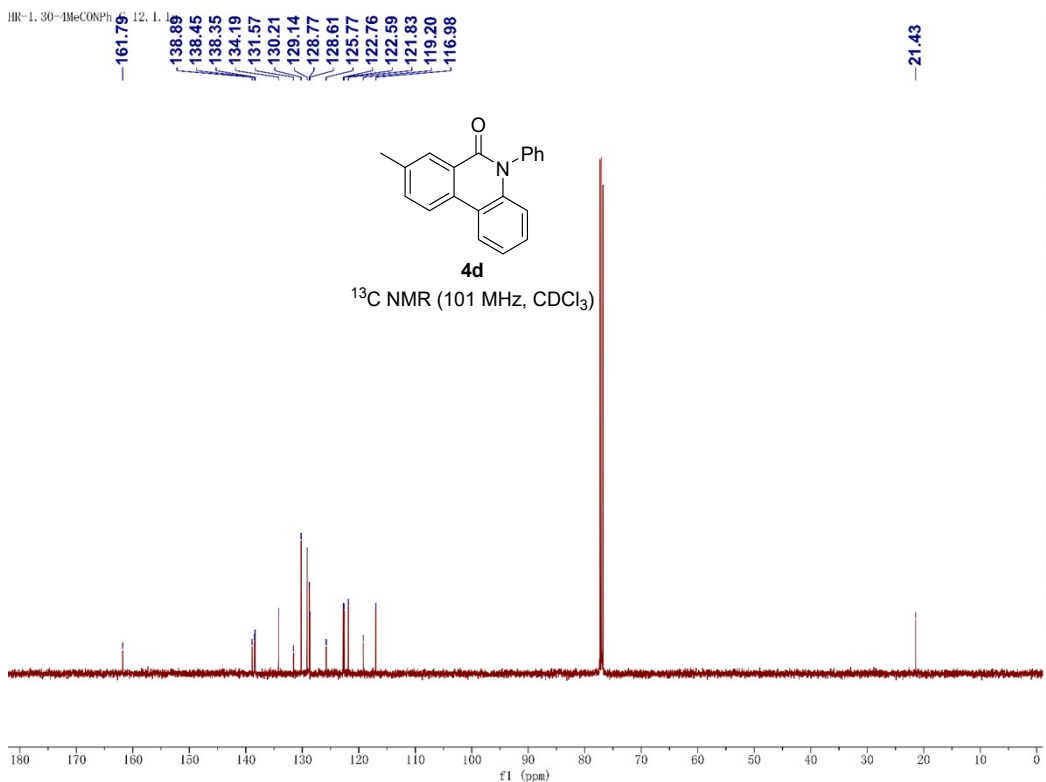


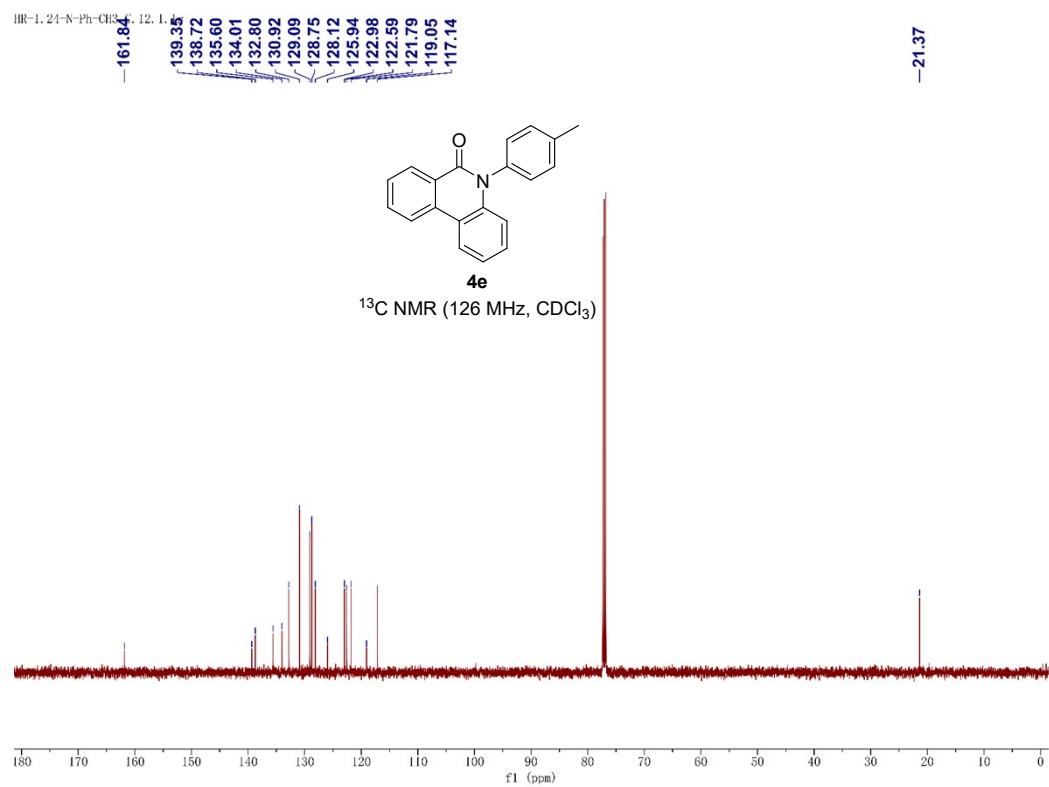
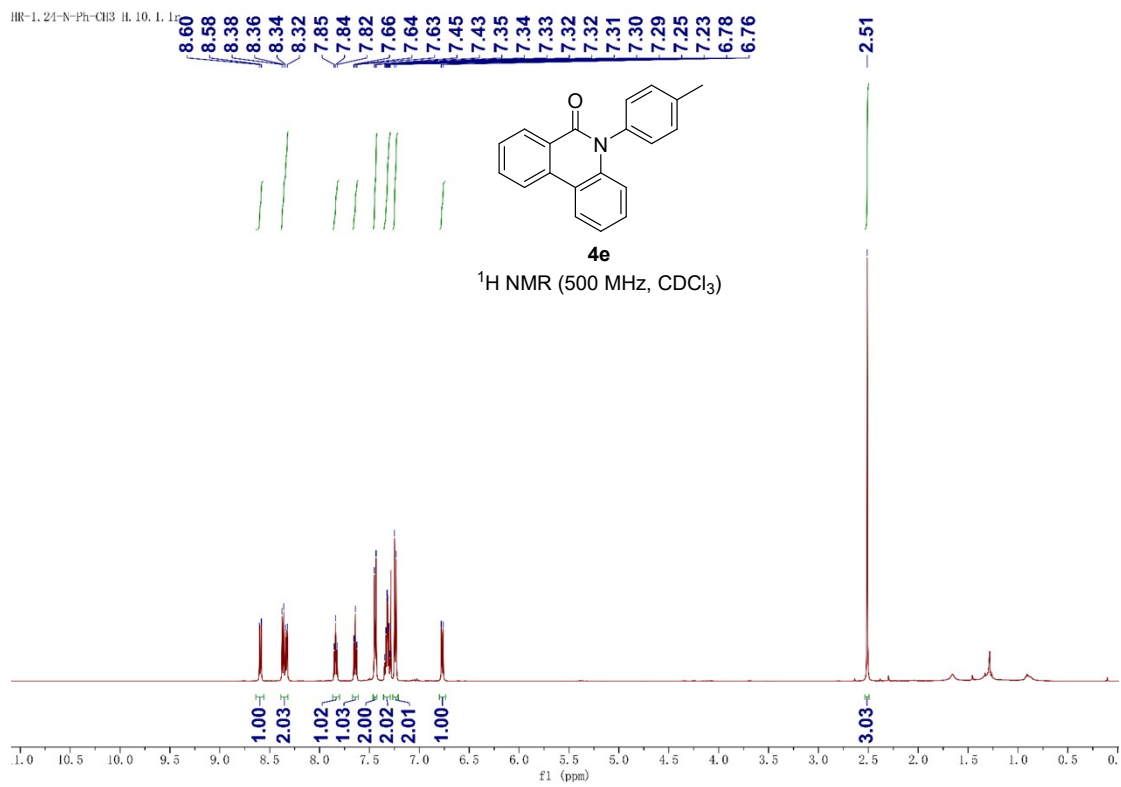


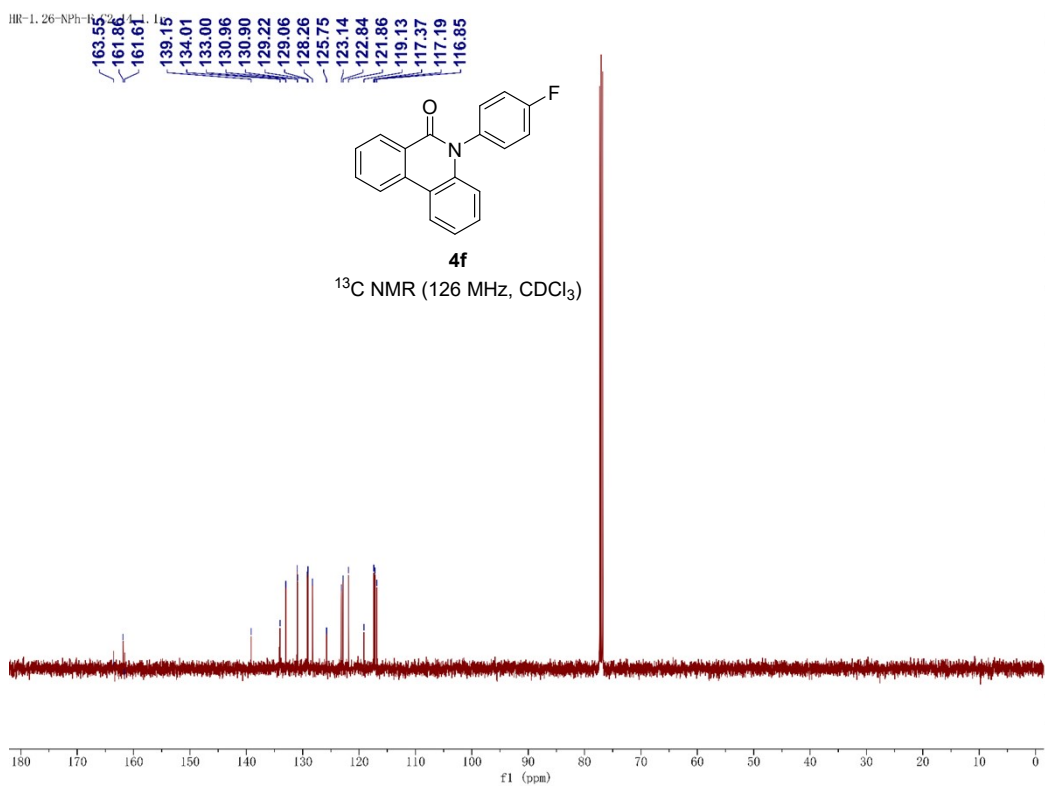
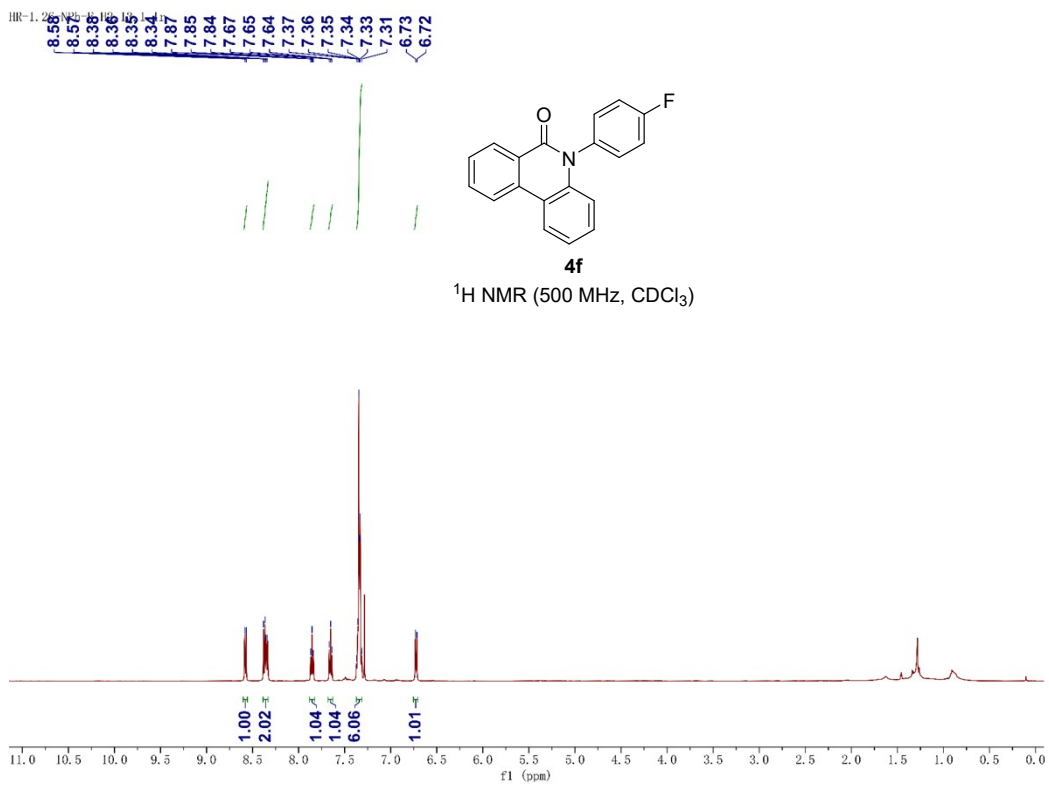
HR-1,30-1MeCONPh H. 10. 1.

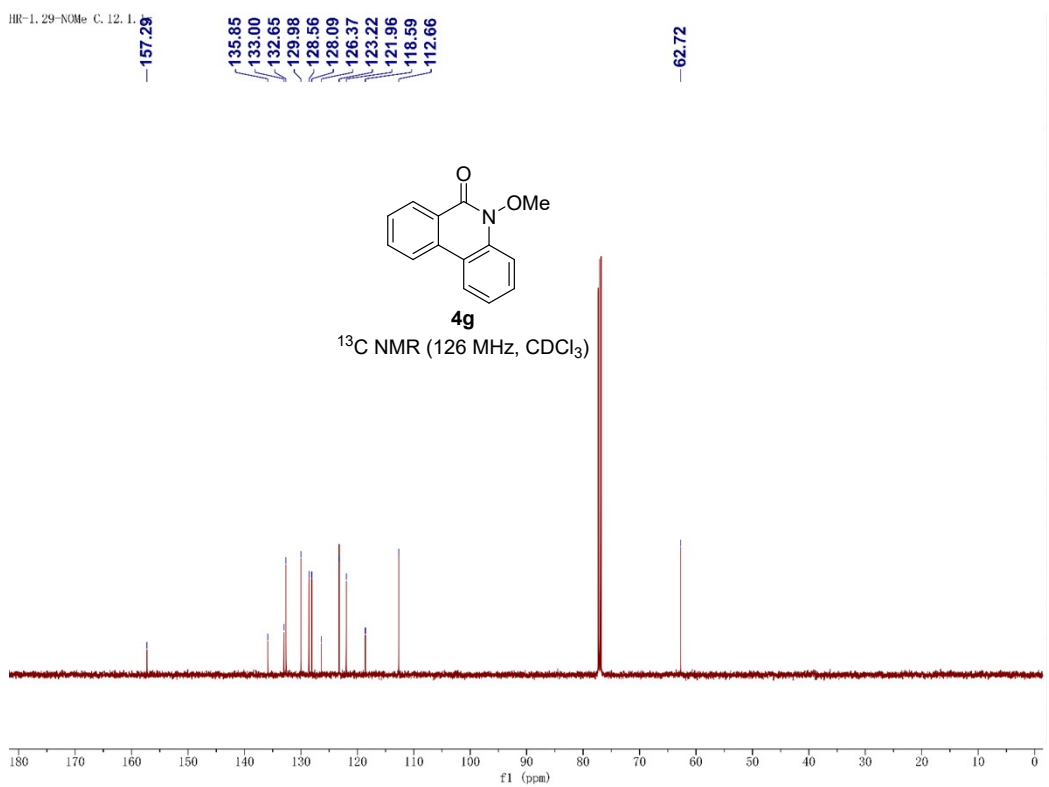
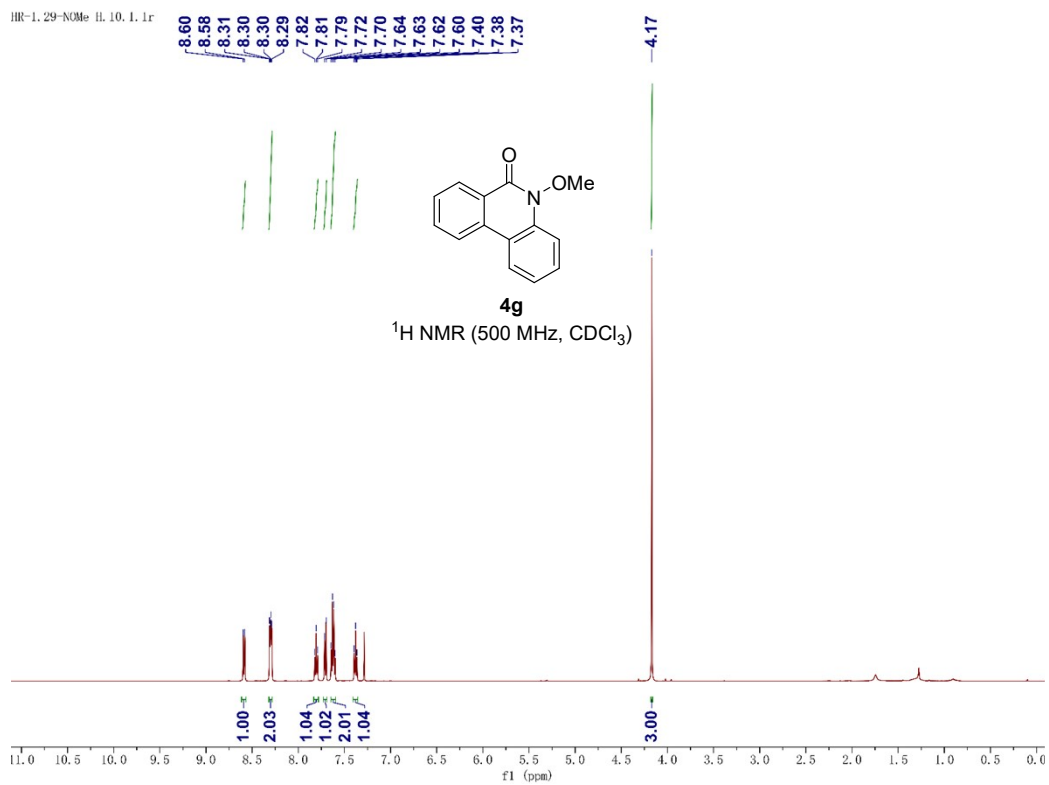


HR-1,30-1MeCONPh 12. 1.

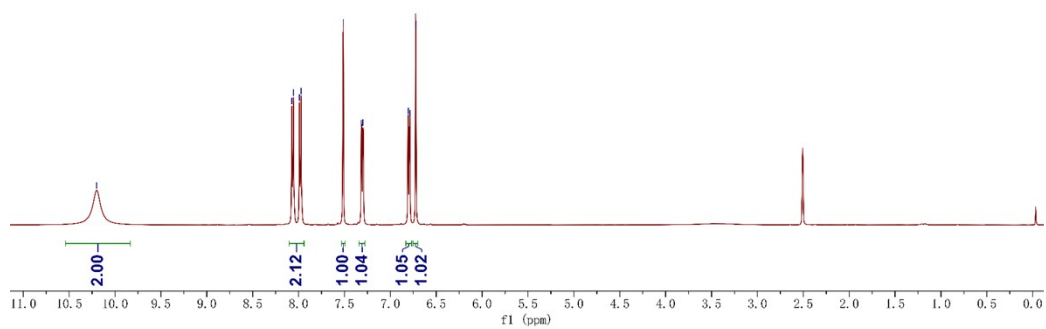
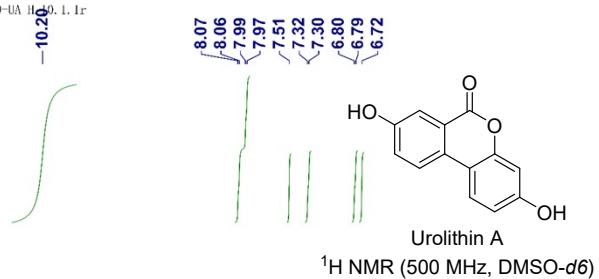




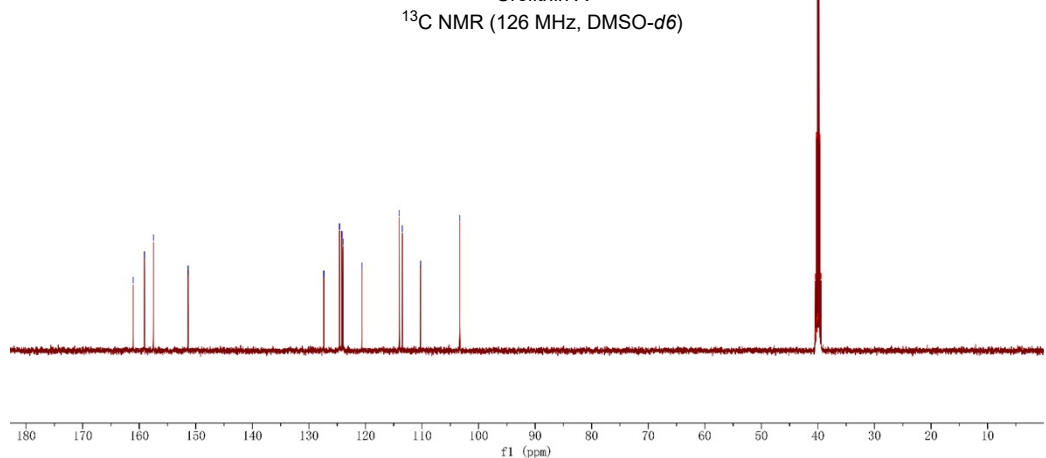
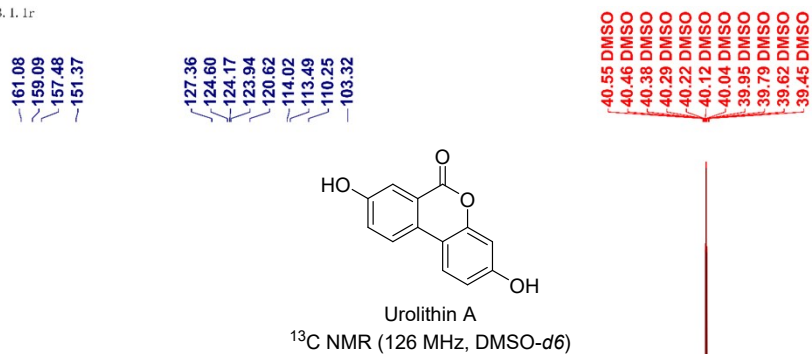




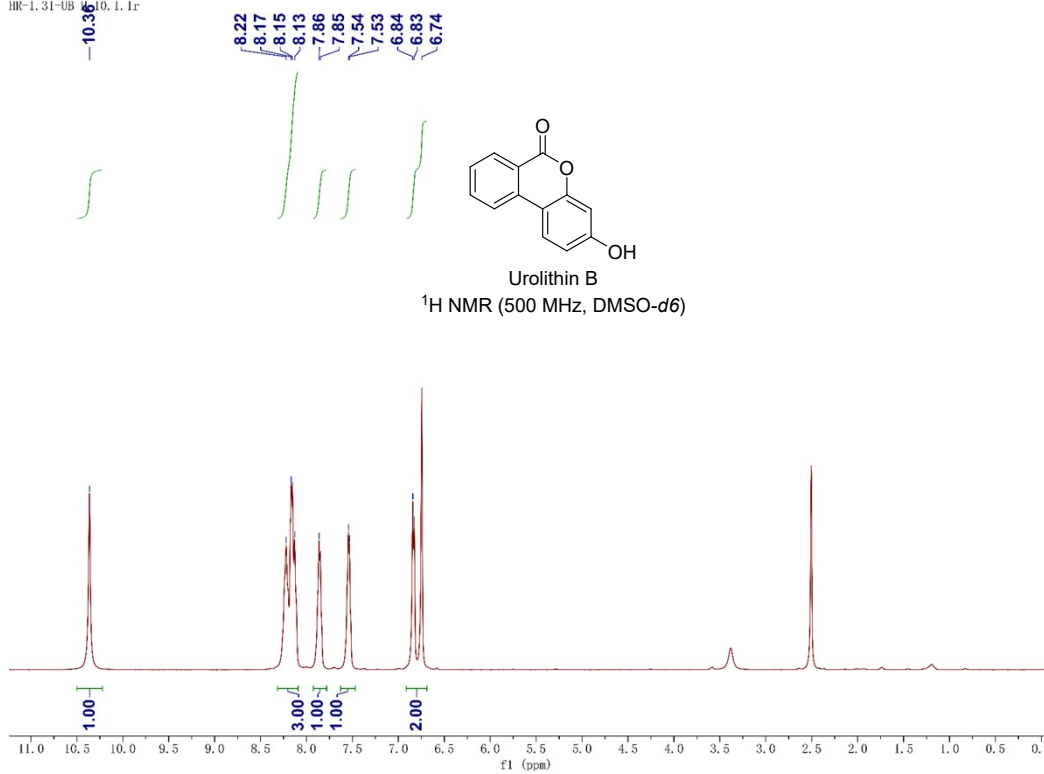
HR-1.29-UA 0.1.1r



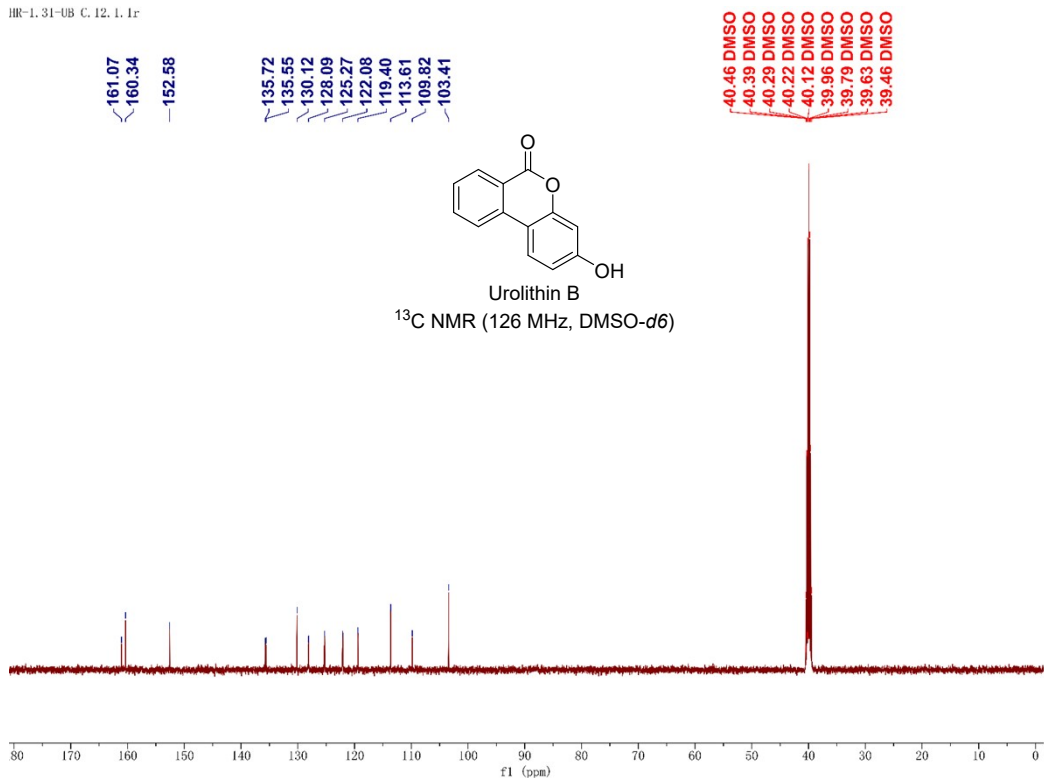
HR-1.29-UA C2.13.1.1r



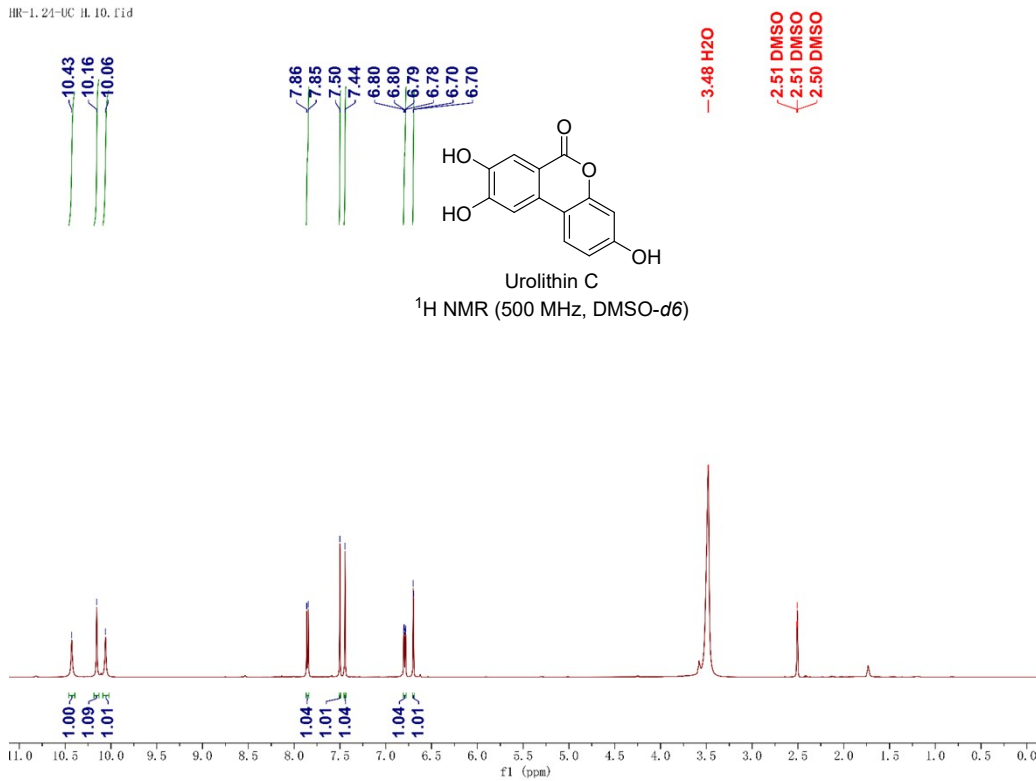
HR-1.31-UB 0.1. Ir



HR-1.31-UB C.12.1. Ir



HR-1.24-UC H. 10. f1.d



HR-1.24-UC C. 10. f1.d

